SEPTEMBER, 1961
Vol. 15, No. 9

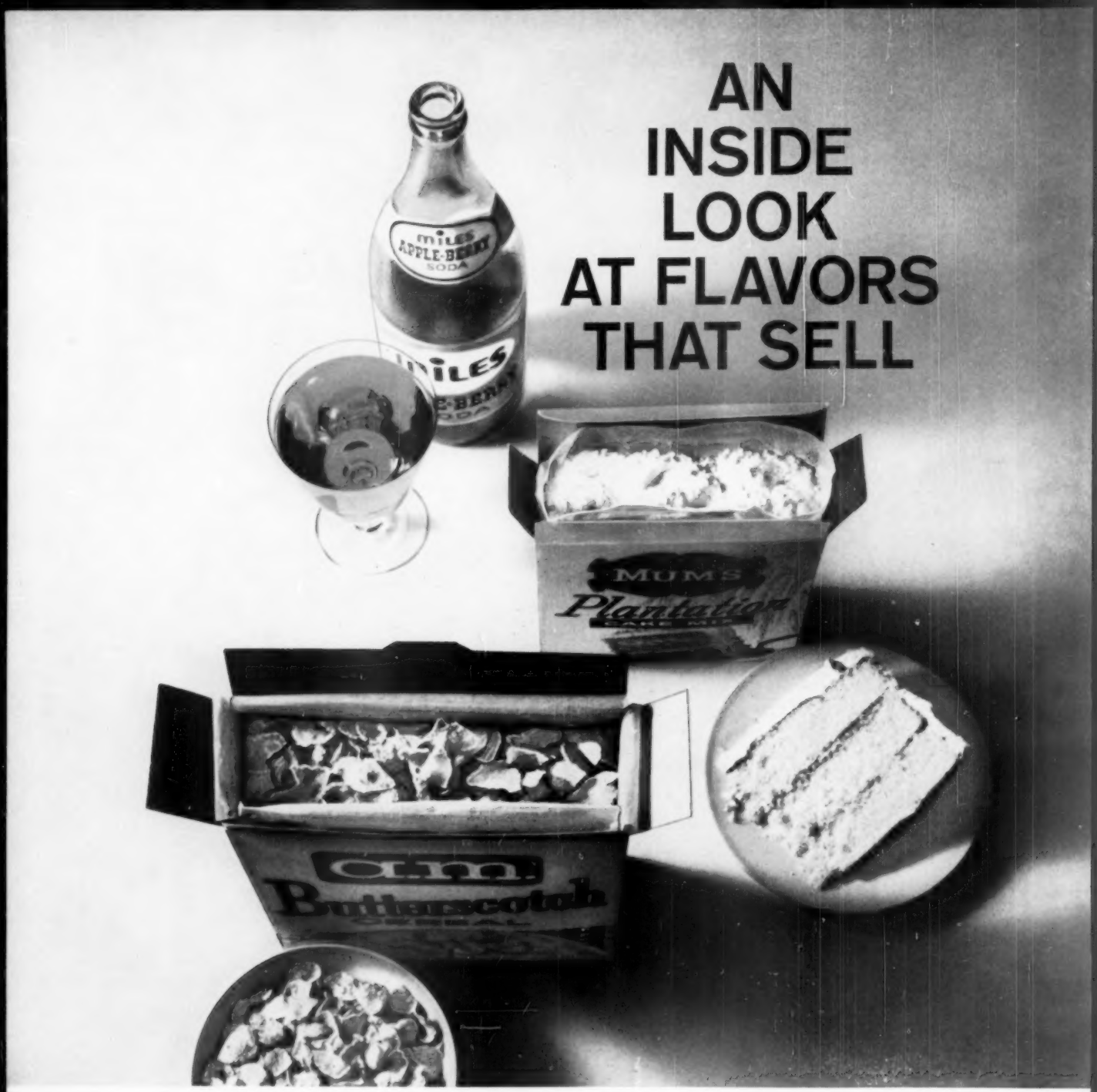
FT food technology

Dickerson | Stone | Tannenbaum | Lebermann

Vilhjalmsson | Bruhn | Galbreath | Higgins

Sinskey | Dobrot | Quirk | Labuza | Roehm

JOURNAL OF THE INSTITUTE OF FOOD TECHNOLOGISTS



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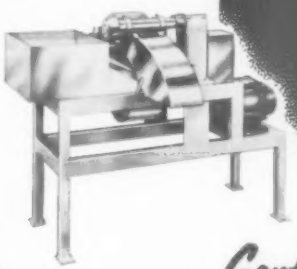

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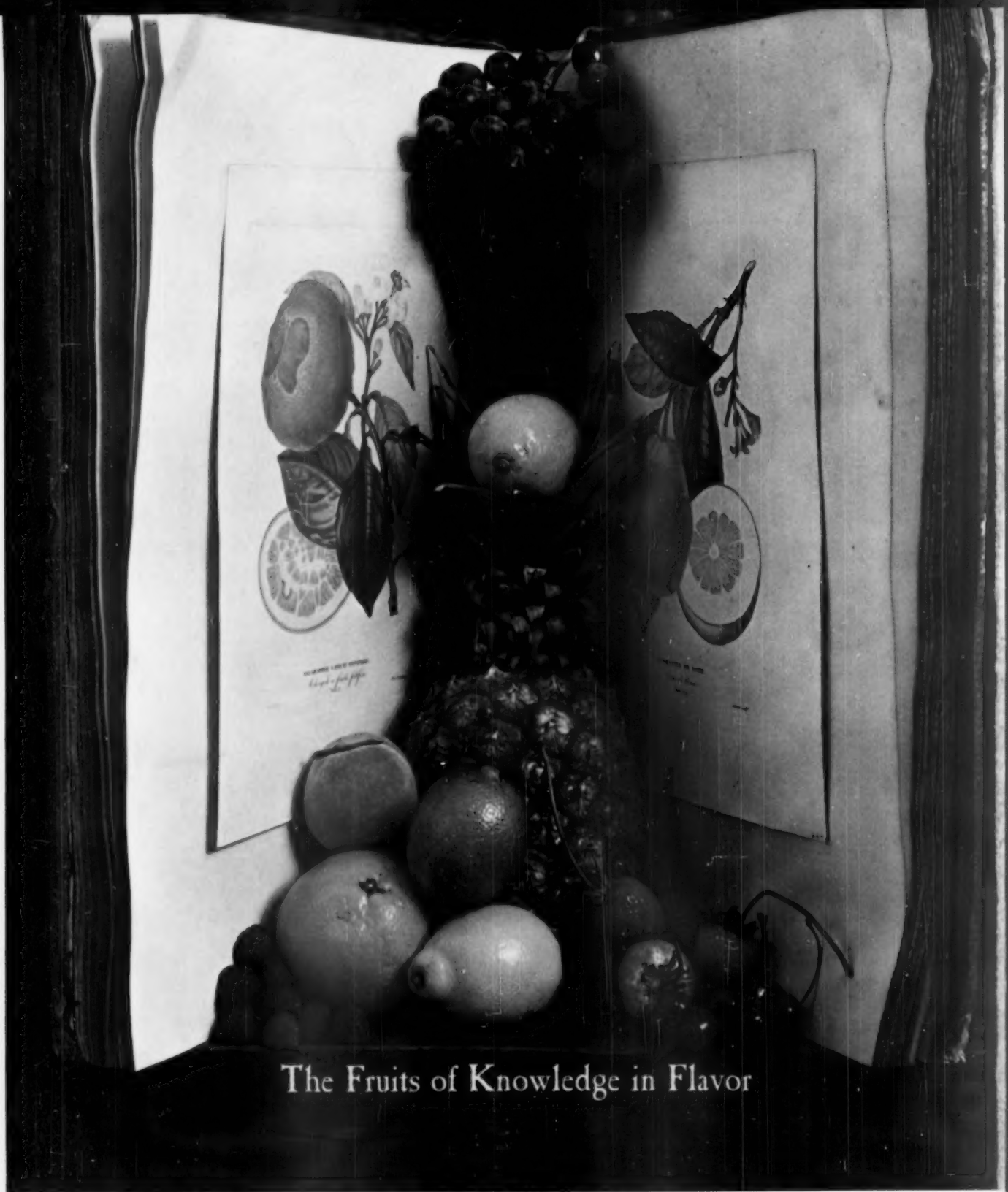

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President Calls on Members to Assert Influence

Should Make Talents Available to Relieve World Food Problems

Dr. Harold W. Schultz
President, Institute of Food Technologists

NOW THAT the historical highlights of the Institute of Food Technologists during its past 21 years have been reviewed in the August issue of *Food Technology*, it seems proper to look ahead and examine the potentials and pending projects of an IFT that has "come of age."

It is very possible that many Institute members are unaware of the many activities, some of them of world-wide scope, now on the IFT agenda.

We are planning for the future, working to make the role and contribution of you, the food technologist, greater and more important in scope and better appreciated by industry and the public. We are attempting to shape the course of IFT so that it will take its rightful place as a force for improving the lot of mankind. We are trying to build up the "image" of the food technologist in the mind of the student and future scientist.

Some projects have already advanced along many lines, because of having been initiated through the foresight of previous officers and councils. Others are being formulated and implemented by committees as outlined in this preview of IFT programs.

Food being as important as it is in many of the world's problems, we, as members of the only world-wide society of food scientists and technologists, should make our talents available and assert our influence more effectively on a broader front. Ways in which this can be done are being analyzed by a new Ad Hoc Committee to Study the Possible Role of the IFT in Assisting Governmental and World-Wide Food Programs. The first assignment for this committee of especially qualified persons, under the chairmanship of Dr. H. C. Diehl, is to explore and define all of the areas where the IFT could, because of its unique membership and objectives, contribute to the solution of important problems involving foods, and recommend procedures for making these contributions.

The Institute's role in helping solve the problems of world feeding involves cooperation with FAO, UNICEF, WHO, ICA, and others. The Food For Peace program of President Kennedy is being enlarged and made a more effective aid to the emerging, freedom-seeking nations of the world, and we, as members of the IFT, can give great assistance to governments in their efforts to help the less fortunate people of the world.

COOPERATION WITH GOVERNMENT

It is a known fact that governments want countries to be self-sufficient. The IFT can help promote such self-sufficiency by giving active aid to food industries in less developed countries. For example, there is a need for a better understanding of quality factors in foods if we are to encourage international trade and exchange. We also need to promote a greater understanding of the use of additives and improved techniques by the food industries of other lands. These and many other subjects may be explored during the First International Congress of Food Science and Technology, in 1962.

This is an ambitious program that offers an opportunity to serve mankind as well as fulfill the objectives of the IFT. Our very participation in attacking broad world-wide problems is bound to improve the professional standing and status of food technologists everywhere.

The Institute will continue its liaison with certain U. S. agencies such as the Food and Drug Administration and the Civil Defense Administration with a view to giving even greater assistance to these agencies in their programs. I am strongly of the opinion that many agencies have problems that our scientifically trained personnel could help solve. And, of course, the U. S. Departments of Agriculture, Health, Education, and Welfare, and Interior all have other programs to which the Institute of Food Technologists can offer real assistance. Comparable agencies in other countries and the United Nations might also be aided.

It should be made clear that we have no plans to dabble in legislative matters, and wish only to render assistance in food programs as a qualified society of food scientists and technologists can do.

INTERNATIONAL CONGRESSES

The IFT is active in promoting international food science and technology congresses through a special committee under the joint chairmanship of Drs. E. M. Mrak and G. F. Stewart. The first Congress will be held in London, England, in September, 1962. It will be sponsored by the Food Group of the Society of Chemical Industry (Great Britain).

ENHANCE PROFESSIONAL STATUS

A Committee on Professional Relations and Status, under the chairmanship of Dr. Gale R. Ammerman, is charged with the responsibility of originating or considering suggestions intended to improve the professional standing and status of food technologists. Personally, I believe that food technologists have both professional standing and high status, but we must promote a greater awareness of that standing and status with management and the public.

A major objective of this committee will be providing more direct service to the IFT membership. As I see it, this can be done by a continuous investigation and collection of data regarding potentials for food technologists, engineers, and scientists, employment patterns, salaries, and enrollment of students studying food technology, food science, and food engineering.

In a second area, there might be included studies of academic and professional improvement programs, employment services, job classifications and descriptions, and ethical and professional behavior.

PLAN TEACHING CONFERENCE

Plans are under way to sponsor a nation-wide conference during the coming year on the teaching of food technology. The importance of such an undertaking seems obvious.

Such a conference is but one of the many activities being planned by the Committee on Education to improve teaching of food science and technology and stimulate student enrollment. Under the chairmanship of Dr. B. S. Schweigert, this group will make the processing industries more aware of their responsibility to get more students into food science and technology. With a few exceptions, colleges and universities have been left on their own to find out how to advertise and promote their curricula in food technology. In order to attract bright young people to the field of food technology, we not only need the cooperation of industry—we need its leadership.

This leadership, this cooperation, should be offered by the food industry as a matter of self-interest. The industry seems not to have recognized that the *need* for scientists and technologists is great—greater than the *demand*, in my belief. If the need is not fulfilled, then the industry may find itself in dire straits.

Food and Agriculture Organization of the United Nations

FOOD SCIENCE ABSTRACTS

Dr. John Ayres is chairman of a new Ad Hoc Committee on Food Science Abstracts. Its immediate objective will be to study all possible ways by which the world's food scientists and technologists can be provided with up-to-date abstracts of all literature important to their field. I feel that the Institute can and should be a leading influence in solving the many problems that exist regarding food science abstracts.

In connection with abstracts, I should express my conviction that the value of the Institute's two journals—*Food Technology* and *The Journal of Food Science*—cannot be overestimated. Many changes are planned for the two leading publicaions. The primary purpose of the changes will be better service for the membership of the Institute, the food industry, and food scientists and technologists throughout the world. The continuing improvement to be seen in forthcoming issues of the journals will be studied and recommended by the Committee on Publications under the chairmanship of Dr. Howard Spencer and will be implemented by Dr. George F. Stewart, Executive Editor.

United Nations Children's Fund

SCHOLARSHIPS HELPFUL

The ten IFT undergraduate scholarships, awarded for the first time to freshmen entering college in the Fall of 1961, are proving to be very helpful in stimulating interest in food technology as a career. Where qualified applications exceeded the maximum number (four) allotted each school, the food industry has been appealed to and has helped get those good students into the department by awarding additional scholarships. This is a most satisfying result.

I believe I should make it clear, however, that it was not just *offering* the IFT scholarships that produced these results—they had to be "merchandised." Where most successful, representatives of the food industry who are members of IFT went from high school to high school with application forms and other material searching out students who were interested—or could become interested—in food technology. Obviously, they found them!

What these "recruiters" did is something that can be done by any Section or any IFT member. A brochure, "Food Industry Careers in Food Technology," is available from the Institute's Chicago headquarters, and this, together with application forms for the scholarships and other information, can be most helpful in inducing students to give consideration to the field of food technology.

The IFT must expand, and is expanding, this program. We must encourage industrial companies to do likewise. There is much we can and will do to tie our program into those of the High School Science Teachers Association, the Future Scientists of America, and similar organizations dedicated to attracting young people to scientific fields. As previously indicated, the Committee on Education is looking into all possibilities.

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GREAT GROWTH POTENTIAL

Because of the great growth of the food industry, and particularly the increasing importance of research and development, I cannot help but feel that the IFT has a great growth potential and will inevitably see its membership increase spectacularly in the next few years. Some members believe we can easily have over 10,000 members. Maybe this is the goal we should set. Whatever the goal is, it can be reached only if we have a carefully planned and executed membership program. Such a program is being studied by the Membership Committee under the chairmanship of Dr. Bruce Morgan.

This year, for the first time, the IFT has embarked on an organized public relations program. With the professional guidance of Merryfield-Wood & Associates, Chicago, the specific projects to be undertaken to achieve certain broad objectives are being determined with the aid of the Committee on Public Relations, under the chairmanship of L. B. Sjöström, and the Executive Committee as well as other key groups. The objectives would be: improving the status and image of the food technologist; promoting the interest and appreciation of industry in food technology; interesting and recruiting students in our profession; alerting the public to the immense contribution of food science; strengthening and expanding the IFT; promoting and publicizing the Annual Meeting; and aiding Regional Sections in their activities. It is expected that some projects will be under way before the end of the year.

World Health Organization

OPTIMISTIC OF FUTURE

Do not underestimate the importance of continuity in planning and carrying out activities of an organization such as ours. Be assured that your President-Elect, Dr. John Jackson, is being constantly informed of the plans contemplated and is given every opportunity to participate fully in the formulation of plans. Not all activities of the IFT can be covered here.

As I view the prospects for the years ahead, I cannot help but be impressed and optimistic. Accomplishments of the past—there have been many many notable ones—provide ample proof that high ideals and important goals can be attained by the IFT. Our potential for service and for growth seems to me to be almost limitless. If we offer and deliver the services of which we are capable, we will reap our own rewards in the form of increased stature and professional standing. We will gain it in the membership growth that is bound to come as potential members gain a greater awareness of the contributions we are making to the welfare of mankind and in the quiet satisfaction that comes from a knowledge that our work has been well done!

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Thirteen Scholars

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Stone | Tannenbaum | Lebermann | Vilhjalmsson | Bruhn | Galbreath

Higgins | Sinskey | Dobrot | Quirk | Labuza | Roehm

Thirteen prospective food technologists are beginning the current school year with financial assistance in the form of scholarships and fellowships administered by the IFT. The awardees will receive a total of $22,000.

Competition for these awards was more intense this year than in any year since the IFT began administering them. Twenty-one undergraduates applied for the eight undergraduate awards, and forty-two applications were received for the five graduate awards.

General Foods Fellowship Awardees, each of whom will receive $4,000, are: Donald R. Dickerson, Charles D. Stone, and Steven R. Tannenbaum.

Donald R. Dickerson will study at the University of Illinois, and plans to follow a career of academic or industrial research. He is married and the father of two children.

Charles D. Stone, who earned his B.S. from the University of Georgia and M.S. from Florida State University, will return to Georgia, where he has been accepted as a prospective candidate for a Ph.D. in food technology. He is married, and a member of several honorary and professional organizations including the IFT.

Steven R. Tannenbaum, a native of the metropolitan New York area, will continue studies at the Massachusetts Institute of Technology, where he has been studying for the past several years. He is married, and plans to finish work toward a Ph.D. He is a two-time winner, for he received the Florasynth Fellowship for the 1960–61 school year.

Kenneth W. Lebermann, as the 1961-62 Florasynth Fellowship Awardee, will return to the University of Illinois, where he completed requirements for a B.S. After earning his B.S., Lebermann completed studies for an M.S. at the University of California. He is another individual familiar to many IFT members, for he was winner of a 1958–59 Gerber IFT Undergraduate Award and was last year's Samuel Cate Prescott Fellowship Awardee. Each of Mr. Lebermann's awards has been valued at $1,000.

Sverrir Vilhjalmsson, a native of Iceland, has been awarded the $1,000 Samuel Cate Prescott Fellowship for the coming school year. He attended schools in Iceland, but earned his B.S. from Cornell University. Since then he transferred to the University of California, where he will study under the fellowship. On completion of his studies he plans to return to Iceland and work in the area of food research or food processing.

Four students are beginning a second year of studies under the auspices of Gerber IFT Undergraduate Awards. Each award is valued at $1,000. Eligibility is restricted to outstanding undergraduates who have achieved at least Junior standing and are pursuing studies leading to a Bachelor of Science degree in food technology, food science, or food engineering. It is of interest to note that each of the four

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individuals who earned a renewal has expressed an interest in continuing studies upon completion of requirements for a Bachelor of Science degree.

Gerber IFT Undergraduate Award renewal plaques for 1961–62 go to John C. Bruhn, Michigan State University; John W. Galbreath, University of Georgia; Lynn W. Higgins, University of California; and Tony J. Sinskey, University of Illinois.

Two new names are added to the Gerber list for the coming school year. They are Johanna M. Dobrot, who will study at Oregon State University, and Leo W. Quirk, who will study at the University of Illinois. Miss Dobrot, a native of Oregon, is active in several campus organizations. She plans to continue studies in food technology until acquiring a Master of Science degree in the subject. Quirk was born in Urbana, Illinois, and has completed two years of active duty in the Army. After earning a B.S., he also plans further study as a graduate student.

Theodore P. Labuza, 1960–61 Dodge & Olcott Scholarship winner, has been named 1961–62 winner of the $1,000 award. He has been placed on the Upper Dean's List on several occasions and will continue studies at the Massachusetts Institute of Technology.

Jeffrey N. Roehm is the 1961–62 winner of the $1,000 Alexander E. Katz Scholarship. Upon completion of requirements for a degree, he anticipates either further study or entering the area of management within the food industry. Jeffrey is attending Oregon State University.

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Enriched FLOUR[1]	2.0	2.5	1.2	1.5	16.0	20.0	13.0	16.5
Enriched FARINA	2.0	2.5	1.2	1.5	16.0	20.0	13.0	*
Enriched MACARONI & NOODLE Products[2]	4.0	5.0	1.7	2.2	27.0	34.0	13.0	16.5
Enriched CORN MEALS	2.0	3.0	1.2	1.8	16.0	24.0	13.0	26.0
Enriched CORN GRITS[3]	2.0	3.0	1.2	1.8	16.0	24.0	13.0	26.0
Enriched Milled WHITE RICE[4]	2.0	4.0	1.2**	2.4**	16.0	32.0	13.0	26.0

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* No maximum level established.

** The requirement for vitamin B_2 is optional pending further study and public hearings because of certain technical difficulties encountered in the application of this vitamin.

1 Vitamin D and calcium may also be used. In enriched self-rising flour, calcium is required between limits of 500-1500 milligrams per pound.

2 Levels allow for 30-50% losses in kitchen procedure.

3 Levels must not fall below 85% of minimum figures after a specific test described in the Federal Standards of Identity.

4 The Standards state that the rice, after a rinsing test, must contain at least 85% of the minimum vitamin levels. The Governments of Puerto Rico and the Philippines also require this rinsing test. If the method of enrichment does not permit this rinsing requirement to be met, consumer size packages must bear the statement, "Do not rinse before or drain after cooking." Rice enriched by the Roche method will meet the rinsing test. The South Carolina law does not require a rinsing test on packages of less than 50 pounds, as the rice in small packages is presumed to be sufficiently clean.

The maximum and minimum levels shown above for enriched bread, enriched flour, enriched farina, enriched macaroni, spaghetti and noodle products, enriched corn meal and corn grits and enriched rice are in accordance with Federal Standards of Identity or State laws. Act No. 183 of the Government of Puerto Rico requires the use of enriched flour for all products made wholly or in part of flour, including crackers, etc.

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New Continuous Production Facility for Processing "Instant" Precooked Beans

W. R. Dorsey, S. I. Strashun, R. L. Roberts
Vacu-dry Co., Oakland, California
and
K. R. Johnson
Quartermaster Food and Container Inst., Chicago, Illinois

SCIENTIFIC ADVANCES in the fields of nuclear energy and missile technology have had unprecedented effect on basic U. S. military concepts. Strategy is now based on highly armed, relatively small combat teams capable of swift mobility and rapid dispersion. These teams must be able to carry all their fighting equipment and subsistenec supplies, and be self-sufficient for extended periods.

Because of the change in military tactics, new feeding systems have had to be developed. Surveys showed that dehydrated foods seemed best suited to the problem. Concentrated in form, light in weight, and easy to ship and carry, dehydrated foods can be designed to give maximum utility for individual or group feeding systems. They can be used alone or as components of compact, ready-to-eat meals. At the same time, they do not require refrigeration, and they have excellent storage stability under all climatic conditions.

The ration-dense properties of dehydrated foods have proven especially valuable to the growing U. S. nuclear submarine force in terms of savings in space, weight, and refrigeration. These advantages also apply to other branches of the Armed Forces, and are intrinsic to dehydrated foods.

There is an equally great potential for dehydrated foods in civilian use, and they are destined to play a key role in the growing field of convenience foods.

The Industrial Preparedness Study on Precooked Dehydrated Legume Vegetables (Lima, Navy and Small Red Beans) reported here was initiated by the U. S. Quartermaster and Container Institute to develop new feeding systems to meet changing logistical requirements.

The Study had it origin several years previously in a research program aimed at developing "instant" precooked beans for use in operational rations. The preliminary investigations were reported in FOOD TECHNOLOGY, Nov. 1956, in "Preparation and Evaluation of Precooked Dehydrated Bean Products," by C. Feldberg, N. W. Fritzsche, and J. R. Wagner. These investigators found that precooked beans split wide open immediately upon their introduction into an air dehydrator. This splitting is known as "butterflying."

"Butterflying" is caused by the differential rate of drying between the skin and cotyledon. The skin dries rapidly and contracts. The slower-drying interior develops vapor pressures of such magnitude as to rupture the skin and produce the "butterflying" effect.

In the study reported here a freezing step was inserted between the precooking and dehydration operations to overcome "butterflying." This proved effective in reducing the degree of splitting in the beans.

Because the freezing step added extra costs in handling and processing, the problem was then approached by introducing high-humidity dehydration conditions during the initial drying stages. It was hoped that high humidity would retard the rapid drying of the skin, helping maintain its initial flexibility, and that when sufficient internal moisture had evaporated the internal forces would be reduced to a level where skin rupture would not occur.

COMMERCIAL FEASIBILITY STUDIED

The commercial feasibility of preparing both precooked dehydrated lima and navy beans by the prefrozen and high-humidity techniques was the objective of two Industrial Preparedness Studies for each of the beans. The studies were made by Vacu-dry Company and submitted to the Government in 1959. The results with navy beans were projected to, and tested on, small red beans. Each study discussed manufacturing processes, equipment requirements, operational considerations, personnel needs, and costs. Data for use in establishing specifications for each legume were also provided.

IMPORTANT CONCLUSIONS REACHED

Some of the more important conclusions reached in the studies were:

1) Navy and small red beans should be blanched before soaking.

2) The High-humidity Dehydration Process was unacceptable.

3) The production of precooked dehydrated legume vegetables by the pre-frozen technique is commercially feasible.

4) The production cost of precooked dehydrated beans can be substantially reduced if the complete operation, including the freezing phase, can be conducted at the dehydration plant.

BLANCHING BEFORE SOAKING

Vacu-dry Company introduced the blanching step primarily to ensure complete rehydration of navy and

red beans in the subsequent soaking operation. Prior to the use of this technique, the degree of rehydration was variable.

The blanching operation also produced a product with better storage stability than in unblanched beans. This provided a clue to solving a problem of great concern to the Institute. The principal difficulty encountered with precooked dehydrated navy and small red beans was rapid development of an unacceptable flavor and odor within 2 to 3 weeks at 100°F. The deterioration was presumed to be oxidative in nature and was attributed to the absorbed oxygen tightly held by the beans even when subjected to vacuum or nitrogen packing conditions.

The blanching operation revealed the possibility of lipoxidase enzyme activity in the rapid deterioration process. Research by the Chemistry and Microbiological Branch, Food Division, Quartermaster Food and Container Institute, revealed that this was actually the case. The results of this research were published in the December 1960 *Activities Report*, a quarterly publication of the Institute, in a paper titled "Enzyme-Catalyzed Oxidation in Legumes—The Implications of Lipoxidase Activity," by Robert B. Koch and Bruno Gini.

As a result of these findings, navy beans and small red beans are now routinely blanched for 15 minutes in atmospheric steam prior to soaking.

HIGH-HUMIDITY PROCESS UNACCEPTABLE

The High-humidity Process is considered unacceptable because:

1) "Butterflying" was not sufficiently reduced to warrant its adoption.

2) The drying time was prolonged.

3) The use of live steam raised havoc with the structural components in the first compartment of the dehydrator.

Fig. 1 shows a cross section of a Proctor-Schwartz Dehydrator similar to the one used in the study.

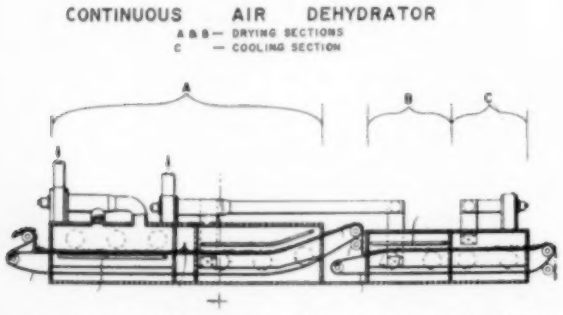

Fig. 1. This dehydrator is similar to that used in investigating the high-humidity dehydration process for "instant" precooked beans. The process proved unacceptable.

High-humidity conditions were obtained by injecting live steam into the air stream of the first compartment. The degree of humidity was achieved both by recirculating part of the air within the compartment, and by metering the amount of steam injected into the air stream. A hygrometer controller activating the live steam line was used to obtain the desired relative humidity of 70% at 163°F.

Dehydration time within the compartment under high humidity was 15 minutes. Time was set by controlling the rate of belt movement. This rate was identical for the second compartment because it was serviced by the same belt.

The rate of speed of the second belt (Fig. 1) could be reduced, but when reduced the belt loading was increased from an initial 1½ inches deep to about 6 inches deep. This depth slowed drying.

Drying time was about 6¾ hours (Fig. 2), and the production rate of finished product was about 500 pounds per hour.

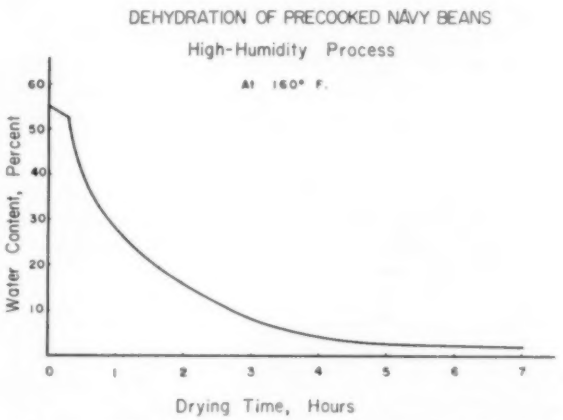

Fig. 2. Curve shows length of drying time for the high-humidity process in the dehydration of "instant" precooked navy beans. The drying-time factor was one of the reasons the process was not considered acceptable.

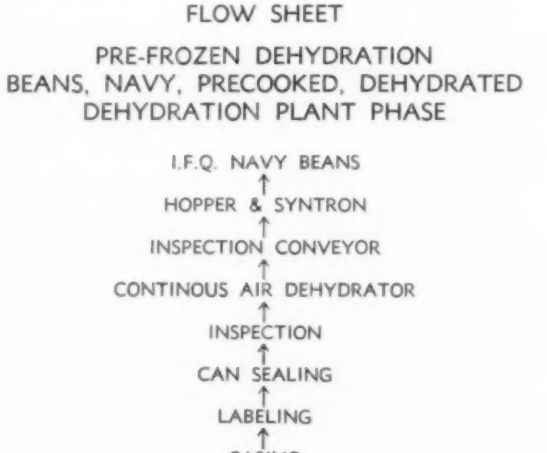

Fig. 3. The production of precooked dehydrated beans by the pre-frozen technique proved commercially feasible. Figure shows the process used.

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PRE-FROZEN TECHNIQUE COMMERCIALLY FEASIBLE

The production of precooked dehydrated beans with the pre-frozen technique was found to be commercially feasible. Fig. 3 shows the process used. The beans were received at the plant in a precooked, individually quick-frozen condition. They were simply metered into the dehydrator at a rate of 2000 pounds per hour. Finished-product rate was about 1000 pounds per hour.

Fig. 4 shows the drying cycle. The beans dried to about 4 percent moisture in about 4 hours at 163°F.

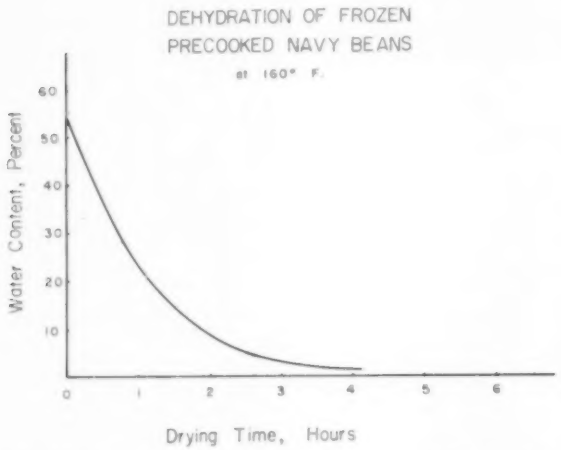


Fig. 4. The drying cycle using the pre-frozen technique in the production of "instant" precooked navy beans. This method substantially reduced the degree of "butterflying."

One of the most important advantages of the pre-frozen technique was that the degree of "butterflying" was greatly reduced. Upon subsequent rehydration of the end product, the beans resumed their normal cooked shape and the splitting was barely visible.

REDUCED PRODUCTION COST

The cost of production of precooked dehydrated beans can be reduced substantially if the complete operation, including the freezing phase, can be conducted at the dehydration plant.

In the studies reported here, the individually quick-frozen beans had to be prepared at a freezing plant because the dehydration plant used in the studies was not equipped with freezing facilities. This created a serious production scheduling problem.

The freezing plant had to perform all the preliminary operations prior to freezing. Though it had the facilities for blanching, soaking, and freezing, they were not in the correct physical location to each other. The freezing plant, in turn, did not have pressure cooking equipment, so this had to be supplied by the dehydration plant.

A special line had to be installed to include all of the processing equipment in its correct sequence, and modifications had to be made to ensure uniform product flow at realistic rates.

It was virtually impossible to superimpose this task upon the full production load at the height of the harvest season. This meant that the beans had to be processed through the freezing stage during lulls, or during the off-season, and put in frozen storage against the needs of the dehydration plant.

Conducting the total operation from dry bean to finished product under one roof would permit better control and greater flexibility of operation, and, most important, reduce costs of transport, frozen storage, installation, double overhead, and extra processing.

LIMA BEANS A SPECIAL PROBLEM

Lima beans created a special problem. In this study, the fresh bean was required instead of a dry bean, as with small red and navy beans. This tied the limas right into the harvest season.

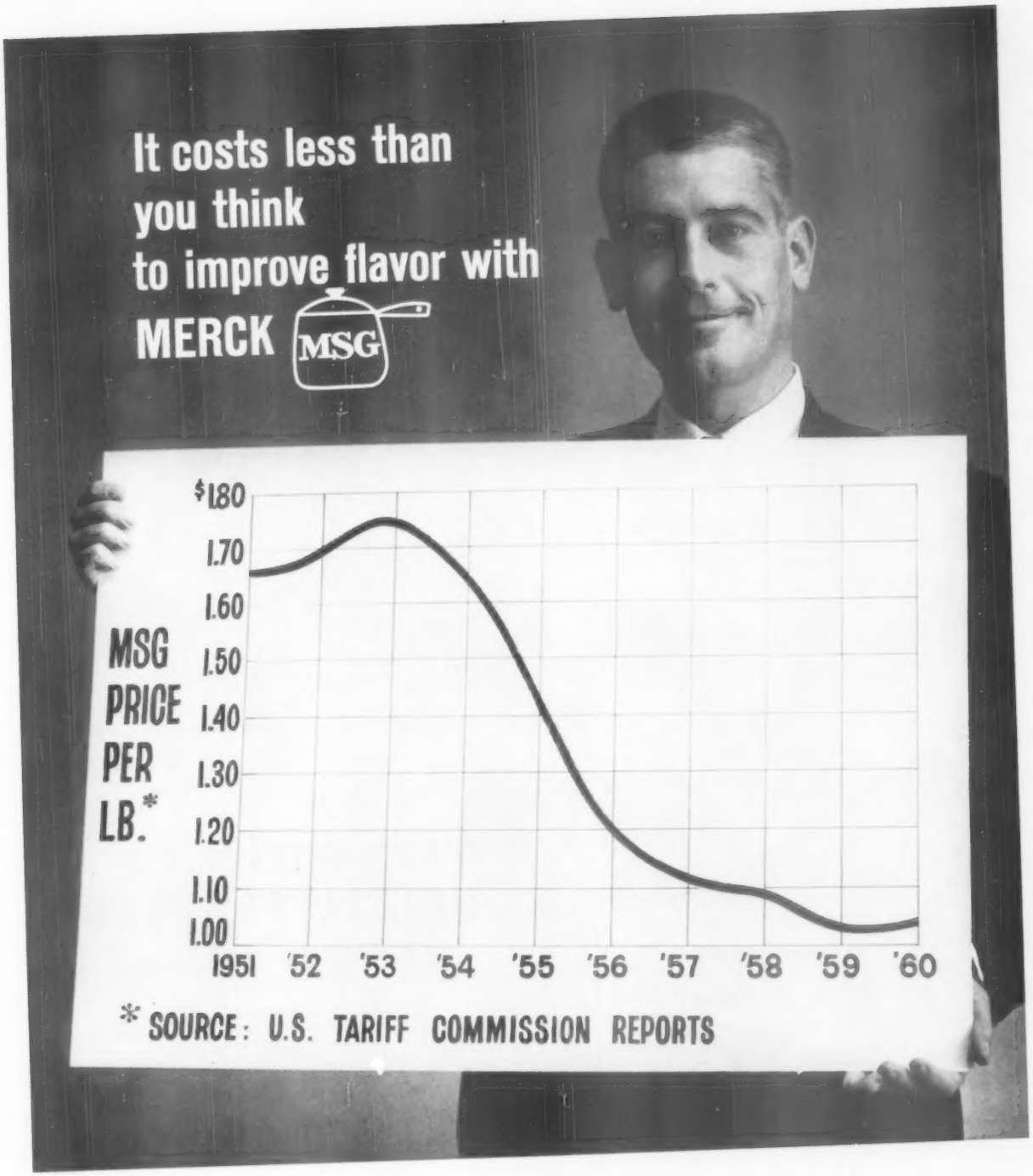
In addition, specifications called for only small lima beans, 30/64 to and including 34/64 inch in width, or medium beans, 34/64 to and including 38/64 inch in width. The dehydration plant was thereby barred from direct acquisition of lima beans from the grower, because it had no outlet for over- or under-sized beans; and this meant that any economic loss resulting from this lack of an outlet would have to be absorbed by adding to the cost of the beans that met specifications. To meet these specifications, the dehydration plant must turn to the frozen lima bean packer for its source of fresh beans.

The frozen lima bean packer grades by quality but does not normally size-grade his beans. However, he can size-grade them by prior arrangement. Moreover, he has channels for selling the beans that do not meet size specifications.

Since the frozen lima bean packer blanches his product, it is fairly easy to extend the blanching operation to a full cook. That can be done without any changes in the processing, and usually without sacrificing rate of through-put.

Lima beans, unlike small red and navy beans, have to be sulfited before dehydration. The best time to do this is right after the cooking operation and before freezing. The packer will initially require some assistance from the dehydration plant to ensure that the right concentration of sulfur dioxide is added to the beans. It can be applied in the form of a sulfite spray as the beans pass over a belt.

By following the procedures outlined above, the dehydration plant will receive from the frozen lima bean packer individually quick-frozen lima beans properly sized and prepared and ready for dehydration. It will then be merely necessary to meter the frozen beans into the dehydrator without any additional preparation at the dehydration plant.



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PRODUCTION SYSTEM IN OPERATION

In keeping with the primary purpose of the Industrial Preparedness Study, the Vacu-dry Company has developed a practical system for mass production of precooked dehydrated legume vegetables at its plant in Selah, Washington.

The system is geared to a rate of 2000 pounds of individually quick-frozen beans per hour, and is capable of continuous operation.

Vacu-dry Company installed, solely for legume vegetables, a special preparation line integrated with an existing dehydration line. Fig. 5 shows the processing equipment, and the order in which it is installed. It is available for large-scale commercial production of "instant"-type beans.

The Vacu-dry installation can also produce simmer-type beans for soup, casserole, and similar uses. It is currently being tested for fresh green peas and whole-kernel corn. Other vegetables will also be evaluated.

Fig. 5. The Vacu-dry system for mass production of "instant" precooked dehydrated legume vegetables in operation at the company's Selah plant is geared to a rate of 2,000 pounds of individually quick-frozen beans per hour under continuous operation. Figure shows the equipment used, and the order in which it is installed.

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Food, History and Problems

THE LETTER informing me that I was the recipient of the Babcock-Hart Award was most welcome.

As Chancellor of the Davis Campus of the University of California, I now find little time to consider the details of the morphology of the yeast cell. I miss this. On the other hand, as a chancellor I am confronted with equally intriguing problems. These relate to the development of a rapidly growing campus, together with other problems of importance to the state, and even to the nation.

Because of my interest in agriculture and foods, I have recently been confronted with experiences that have further aroused my interest in humanity and subsistence. I have been concerned with the zeal of misinformed people who would prohibit the use of all agricultural chemicals, synthetic fertilizers, and food additives—ostensibly for the safety and welfare of man.

These people are unaware that scientific revolutions are not reversible. They are uninformed on the four great revolutions caused by man, and the results of these revolutions on populations, subsistence, and the welfare of man. I would like to discuss these briefly, and then lead into the more detailed problem relating to the use of chemicals in agriculture and food.

It is apparent that the nutrition of early man was anything but ideal. He suffered from the environment surrounding him. Food was scarce. When food was obtained, it was perhaps difficult to eat because it was uncooked.

The discovery of the use of fire changed this situation. It resulted in one of the great revolutions in the history of man. This occurred long before the dawn of history and was of such great importance because it overcame problems relating to comfort and the edibility of foods. It improved the acceptance of both plant and animal foods by tenderizing them. It enabled man to use materials that, uncooked, would be quite indigestible or difficult to eat. More than this, it enabled him to heat his environment and make it more pleasant. And for the first time, he learned something about the preservation of foods by smoking and drying.

The use of fire by early man resulted in an increase in population. One wonders, however, if even in those days there may have been naturalists who argued against the use of fire and insisted that man should eat meat only raw—or even spoiled.

The second great revolution in the history of man occurred with the discovery of practical agriculture. This took place 10 to 15 thousand years ago, in the Neolithic period. Naturally, it did not come all at once. Most great developments occur slowly.

The great advantage of agriculture to early peoples was that it enabled them to have plant and animal foods more regularly. They could now settle in one place permanently and no longer have to live a nomadic life in a search for food.

The development of agriculture also resulted in an increase in population. Here again, one wonders if there might have been ancient naturalists who in the early days of agriculture objected to it and preferred to abide by the old system! In any event, agriculture, a scientific discovery, has remained and improved ever since.

The third great revolution was the development of cities. This occurred about 6,000 years ago, and seems to have taken place about the same time in a number of countries, including Egypt, Iraq, China, and Mexico.

Close association in cities enabled the division of labor and establishment of stores of food, and hence the possibility of relieving local shortages through the regular operation of trade. Once again, the change resulted in a great increase in population. But there must have been those who objected very seriously to the development of the cities. As a matter of fact, we still have such people.

The fourth great revolution is still taking place. In consequence, it can easily be overlooked. Most certainly we are not in a position to have perspective so that we may compare the effects of the present revolution, with those of the past. The present one is the scientific revolution, which has brought about greater changes than any of the others. As a result of discoveries of the fundamental nature of the world, man has been able, intentionally and deliberately, to alter his way of life. The great areas of particular interest to this meeting are those concerned with environmental health, and particularly those of food supply and safety.

It would be impossible in this brief paper to record the enormous benefits that this revolution, the fourth in the history of man, has brought to man. In the past 50 years the terrible inroads caused by such epidemics as smallpox, the Black Death, and typhus have been eliminated. Famines no longer occur to the extent to which they did. We are now concerned primarily with degenerative diseases such as cancer and heart trouble. In this country, we have gone from the problem of underweight to overweight. We have increased the food supply in many parts of the world, and have literally wiped out the nutritional deficiencies and famine.

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[a] 1961 Babcock-Hart Award Address presented at the Twenty-first Annual meeting of the Institute of Food Technologists, Statler Hilton Hotel, New York City, May 9, 1961, by Dr. E. M. Mrak, Davis, California.

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The scientific revolution has certainly extended to the farm. Today, the average American farmer produces about 10 times as much food as did the American Indian or African, and with the use of machinery he does it with much less work. The need for farm labor has decreased year by year and is still decreasing. The real problem has been to extend the scientific agricultural "know how" of the United States to the rest of the world. This has not been easy, and will require some time. It is easier and safer to live today than ever before: we have an abundance of foods. The scientific revolution has done more to increase population than any of the others. It has now reached the state where any number of people are wondering about the adequacy of the food supply in the future as well as the adequacy of energy, natural resources, and even space in which to live. Thus far we have shown that Malthus was wrong, but it appears that this is only temporary. We have altered the situation so that the natural forces have had less and less influence on environment, and more and more is determined by social forces established by man. The central part of this great revolution is the discovery that nature can be controlled and conditions modified. Populations can now increase greatly without new discoveries. This is the situation unless we interfere with the changes that are taking place either by failing to meet the needs for food or by controlling the increase in population.

I am concerned in this paper about our failure to meet the needs for foods, and particularly with respect to those uninformed people who would decrease our food production drastically. This would create a situation that Decker stated very aptly, "If we were to adopt a policy of 'let nature take its course' as some individuals thoughtlessly advocate, it is possible the experts would find disposing of 200 million surplus human beings even more perplexing than the disposition of the infamous food surplus."

Kleiber points out that if we meet all our food needs with eggs, we will soon reach the end. On the other hand, if we resort to potatoes, we could get by with an area of .15 acres per person, or about 6 persons per acre. We could feed about 8×10^{12} people. This would be in the year 2800 A.D. if our birthrate continues. This is rather soon as history goes, and is practically right now in terms of the development of man. In an article published in *Science* in November, 1960, Foerster, Mora, and Amiot predicted "Doomsday: Friday, 13 November, A. D. 2026." They even indicate there is a possibility that our great, great grandchildren will not starve to death but will be squeezed to death because of numbers on the earth. Darwin indicates that our population will double in about 40 years, and then the number will be doubled again and there will be over 5 billion people in the world.

In spite of this, I would still like to talk about food. A recent publication of the USDA, titled "Food for the Future—Through Research," indicates that "by 1975 the U. S. will need about 200 million more acres of crop land than now—if yields are to be the same as in 1956." It goes on to point out that we don't have 200 million more acres and won't need them, because of the rising productivity on farms. It indicates there will be better conservation of soil and water resources as basic to the increased productivity. It does not state anything about agricultural chemicals, but I am certain that this is implied.

Some interesting information is presented in an exciting book entitled "The Next Hundred Years," by Brown, Bonner and Weir. These gentlemen point out that there are a hundred thousand new mouths to feed in the world each day. They indicate that 55 per cent of the world has less than 2250 calories per day. About 25 per cent of the world's population has over 2,750 calories per day, and 25 per cent has between 2,250 and 2,750.

In order to live an energetic and satisfactory life, humans require about 3,000 calories per day. To obtain this, the individual must eat the equivalent of about 1.8 pounds of plant material containing .7 pounds of carbon per day.

The United States has more than 2 cultivated acres per person. In North America there are about 3.6 cultivated acres per person, whereas in Japan there is only 1. In the United States the acre yields about 4,500 calories, in Asia about 4,000, in west Europe 7,000 to 8,000, in Japan 13,000, and the world average equals about 3,800 calories per day for the population. This is assuming that diet patterns are quite similar to what they are at present. We eat about one-third of our diet in animal food, whereas the Asian eats about 5 per cent of animal foods, and the European is some place between.

The question may be asked why production in Japan is so high. The answer is the use of intensive agriculture, and particularly fertilizers and, I presume, agricultural chemicals. In Japan they use 56.5 pounds of fertilizer per acre, whereas in the United States we only use 11.8 pounds. This indicates the importance of agricultural chemicals.

Brown and his associates have concluded that if production is maintained at the present Japanese level and consumption is at about 3,000 calories per day the world could support about 6.5 billion people. According to Darwin and other demographers, it will not take very long to accumulate this number of people if the increase continues at the present rate.

Insofar as types of food and food technology are concerned, it is conceivable that eventually we might be compelled to change our diet patterns. At present we grow a great deal of vegetable matter and feed it to animals, and these are then used for human food. The efficiency is between 5 and 10 per cent. As an example, forage grain for steers yields about 43 pounds of protein per acre, silage for milk about 77 pounds, soybeans (as such) about 450 pounds, and alfalfa as grown in the western United States about 1500 pounds. We could very well learn to obtain our protein directly from alfalfa.

It is apparent to me that as time goes on we must learn to increase our production per acre. In order

to do this, we must use more synthetic fertilizers, and certainly agricultural chemicals. That is the situation, regardless of what the organic farmers may say.

There are other ways in which food production can be increased. Naturally, at present there is great wastage of fresh produce. I have seen foods wasted in South America merely because they weren't used fresh, yet they could have been processed.

The politicians are making "political hay" of the surpluses we have on hand. If we believe the figures of the United States Department of Agriculture, perhaps we should think in terms of retaining the surplus for future use. Perhaps the most effective thing we can do now is to learn more about the use of new and more effective chemicals and other means of controlling pests that are apt to destroy these surpluses.

Decker has pointed out so very well that our cultivated crops in North America are attacked by over 3,000 economically important species of insects, many plant disease agents, and unestimated numbers of nematodes, rodents, weeds, and other competitors.

In 1954, the United States Department of Agriculture estimated that to offset the losses of agricultural production as a result of pests, an extra 88 million acres would have to be cultivated and that the losses subsequent to harvest would equal the production of an additional 32.8 million acres. Estimates of destruction caused by agricultural pests, made independently by several other agencies, range between $8 and $15 billion annually. Decker has pointed out that if agricultural chemicals were to be banned tomorrow, the yield of many crops would be reduced 10 to 90 per cent and the surplus stocks would soon disappear. The price of most food items sold in stores would double and some would treble, while still others, notably fruits and vegetables, would totally disappear from the open market. He also points out that in our pastures and meadows today, we frequently produce more insect protoplasm than the animal weight gains per acre. In some pastures it is obvious that unused weed growth exceeds the production of palatable forage. Obviously we are still sharecropping with insects and weeds. So long as surpluses prevail, this is permissible, but the day is not far distant when such inefficiency can no longer be tolerated. This is the day when it will be necessary to increase the use of agricultural chemicals or other means of controlling these insects, regardless of what the organic farmers may say.

The agricultural situation has changed greatly because of intensive cultivation. When the white man came to North America, there were relatively few people here and there were about 2,000 acres per capita. The variety of insects attacking crops was as great in number as it is now; however, agriculture was not so intensive. In the years that followed, the number of crops increased and there was plenty of food for all, including the insects. The farmers raised only feeble objections to sharecropping with insects and other pests. At present we have about ten acres per person, seven of which are classified for farm land but only two of which are devoted to crop production.

The great pressures resulting from the intensification of agriculture are bound to produce problems. Not only is it giving the insect more food and better opportunities for growth, but it even encourages insects to attack crops that they ordinarily would avoid. New insects have been introduced. Many factors have contributed to the increased prevalence of destructiveness of the weeds, fungi, bacteria, nematodes, viruses, and whatnot over the last 50 years. We may as well face the fact that as time goes on we will have to produce more food, and in order to do this we will have to use more chemicals to control these pests.

We have a real problem insofar as the use of agricultural chemicals is concerned. Strong effort is being made to outlaw the use of these chemicals. This might seem ridiculous, but when one experiences, first hand, the forces behind this effort, it does not seem so far removed. In order to cope with this very serious situation, so that we may have good food for the future, it is necessary to counteract this strong force that is influencing both public opinion and some of our legislators.

W. E. Baier has commented on the pros and cons of food additives. He points out that the problem is due to a lack of understanding by some legislators, agriculturists, food producers, and a considerable segment of the consuming public. He also points out that it is a paradox that those people who most seriously want to gain an understanding of the subject often appear to end up with the least understanding and the most misinformation. This is well manifested by those who are motitvated by interest in the subject to read books such as Longgood's "The Poisons in Your Food." Unfortunately, many of these books do not paint a true picture with respect to foods and food additives, but rather one of fear. I believe it is time that we counteract this. Instead of having poisons on our bookshelf, we should replace them with honest and interesting publications. These should be written in a manner that will induce people to read them. I believe it would be possible to prepare such publications that would be as exciting to read as "Microbe Hunters," by de Kruif. This, in my opinion, would be far better than the Madison Avenue approach.

At a food additives conference held in New York last October, Dr. Robert White-Stevens pointed out that, as with all matters of universal concern and vast complexity, ample opportunities are provided for misunderstanding, illogical conclusions, and unfounded fears. This has certainly been the case with respect to the wonderful advances made through the use of agricultural chemicals and food additives.

Dr. William Baier, of the Sunkist organization, pointed out some time ago that Congressman Miller, in his amendment, recognized, perhaps for the first time in all legislative history, this simple but often ignored fact: "there are no harmless substances; there are only harmless ways of using substances." This is an extremely important concept that definitely applies to natural foods as well as those that have been substantially ameliorated. This calls to mind an article that recently appeared in a newspaper, "Dr.

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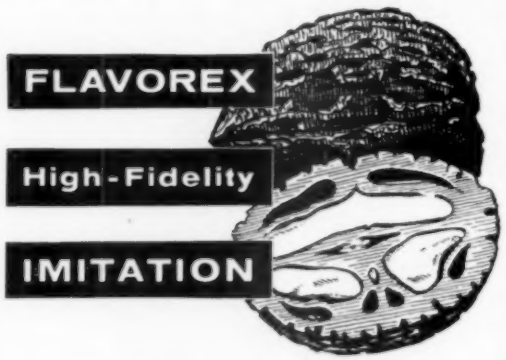

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I believe we need an educational program that covers all segments of life and is multiphasic in approach. It should involve books, pamphlets, brochures, radio, television, and even personal contact. It should be dignified, and it should meet the requirements of the particular individual.

I have seen a great deal of literature on this subject in recent years, but I venture to say that it has had relatively little effect. Most of the money used in this area to educate the consumer has been wasted.

I could continue and cite example after example, but I have already gone a long way around in coming to the point of chemicals and foods. I might well summarize by saying that man is merely an animated mass of chemicals. This is well illustrated by a table formulated by Dr. Lawson and published by Jensen in his book "Man's Foods." The following table indicates that adult man in chemical terms represents enough:

Water to fill a 10-gallon keg
Fat for 7 bars of soap
Carbon for 9,000 lead pencils
Phosphorus for 220 matches
Magnesium for one dose of salts
Iron to make a nail
Lime to whitewash a chicken coop
Sulphur to rid one dog of fleas

This tells the story succinctly. I might add one more point with respect to chemicals, and it is one relating to some work carried on by Decker on the toxicity of chemicals and accumulation of agricultural chemicals in steers. In five separate studies involving the feeding of qualitative hydrocarbon insecticide residues to steers, one or more animals were found to have abscessed livers at the time they were sacrificed. It so happened that in every case these were animals from the check lot, but those that had received the hydrocarbon were completely free of abscessed livers. As Decker points out, the mere suggestion that these materials may have some therapeutic value almost inevitably draws a quick "now you're being facetious." Why? He goes on to state what would happen if the odds of 5 to 0 were reversed; it would certainly be hard on agricultural hydrocarbons.

It seems to me that the whole story is well illustrated in two poems written by Kenneth Boulding—"Conservationist's Lament" and "Technologist's Reply." The lament indicates that the individual's cup is half empty, and the reply that it is half full.

I would rather abide by the technologist's point of view and educate those who do not have the facts.

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Summary of Toxicity Data on Sulfur Dioxide

Floyd DeEds

Chief, Pharmacology Laboratory, Western Regional Research Laboratory,[a] Albany, California

SULFUR DIOXIDE as a gas, or in water solution, or as one of the salts of sulfurous acid is used in the food industries of many countries. The use of sulfur dioxide as a preservative dates back many centuries. It is unique in that it serves a greater variety of purposes than any other single food additive. Sulfur dioxide reduces or prevents microbiological spoilage. Its antioxidant properties retard the loss of ascorbic acid and carotene. Sulfur dioxide inhibits enzyme-catalyzed oxidative discoloration and nonenzymatic browning during the processing, storage, and distribution of a variety of foods.

Despite the long history of successful use of sulfur dioxide in food industries and the existence of data establishing the safety of such use, inquiries as to its safety occur with surprising frequency. It therefore seems appropriate to summarize and comment on toxicity data for sulfur dioxide.

The question of safety in using sulfur dioxide in dried fruits is primarily a problem to be answered by chronic toxicity studies rather than investigations of acute toxicity. Nevertheless, a few data on acute toxicity will serve to emphasize a point pertinent to chronic toxicity.

Hoppe (*J. Pharm. Exp. Therap.* **101**, 101; 1951) found the LD_{50} dose of sodium sulfite injected intravenously in mice to be 175 mg per kilogram of body weight. Flury (*Abderhalden's Hdb.*, 4.7b, p. 1396) reported the acute toxicity of sodium sulfite as follows:

Species	Route	Dose in mg/kg
Guinea pig	Subcutaneous	600
Guinea pig	Intravenous	200
Cat	Subcutaneous	1300–1600
Cat	Intravenous	400
Dog	Subcutaneous	1300–1600

The above data show a marked variation in acute toxicity with species and route of administration. If it is assumed that toxicity for man is the same as for the mouse, the LD_{50} for sodium sulfite given intravenously for a 70-kg individual would be 12.25 g. High-moisture dried fruits have a pH value in the range of 3.5–4.0, and the only way in which sodium sulfite could enter the blood stream after eating such fruit treated with sulfur dioxide would be through intestinal absorption after neutralization of any free sulfurous acid by the alkalinity of the intestinal tract.

The above-mentioned low toxicity of sodium sulfite emphasizes the impossibility of acute toxicity due to ingestion of dried fruits treated with sulfur dioxide, even if 3000 ppm were used. The possibility of chronic toxicity resulting from daily ingestion of such fruits is even more remote, because the small amount of sodium sulfite absorbed is rapidly converted to nontoxic sodium sulfate (Torald Sollmann, "A Manual of Pharmacology," eighth edition, page 153, W. B. Saunders Company; and A. J. Lehman, Association of Food and Drug Officials of the United States, vol. XXI, page 29, January 1957).

The most frequently cited report on the chronic toxicity of sulfites is that of O. G. Fitzhugh, L. F. Knudsen, and A. A. Nelson (*J. Pharm. Exp. Therap.* **86**, 37; 1946). Those investigators used 504 rats in several long-term feeding tests that collectively covered dietary levels of 0.0125, 0.025, 0.05, 0.1, 0.2, 0.25, 0.5, 1.0, and 2.0% of sodium bisulfite. They concluded that the toxic effects observed could be attributed to destruction by the bisulfite of thiamine and possibly other B-complex vitamins in the diet. They interpret their data as showing that dietary levels of at least 1000 ppm of bisulfite, equivalent to 615 ppm of sulfur dioxide, are needed to produce evidence of thiamine deficiency.

In interpreting the significance of this investigation for man, let it be assumed that man's susceptibility to the toxic action of sulfur dioxide is the same as that of the rat, an assumption justified by similar thiamine requirements. Man would then show evidence of thiamine deficiency, and possibly deficiency of other B vitamins, if his daily diet contained 615 ppm of sulfur dioxide. To attain this level of sulfur dioxide intake, man would have to ingest a diet consisting *entirely* of dried fruits containing 615 ppm of sulfur dioxide. This impossible situation emphasizes the importance of the fact that dried fruits treated with sulfur dioxide constitute only a small part of man's diet.

Proper evaluation of the significance of the conclusion that 615 ppm of sulfur dioxide in the daily diet of the rat is deleterious should take into consideration other important factors: 1) the vitamin A, thiamine, and ascorbic acid content of dried fruits; 2) the desirability of comparing the effects of SO_2-treated and untreated dried fruits upon the growth, general health, and longevity of, and histopathological findings on, experimental laboratory animals; and 3) the possibility that untreated dried fruits may cause toxicity due to reaction products formed in deterioration of the untreated fruit.

1) The following table summarizes data on the vita-

(Continued on page 33 following research section)

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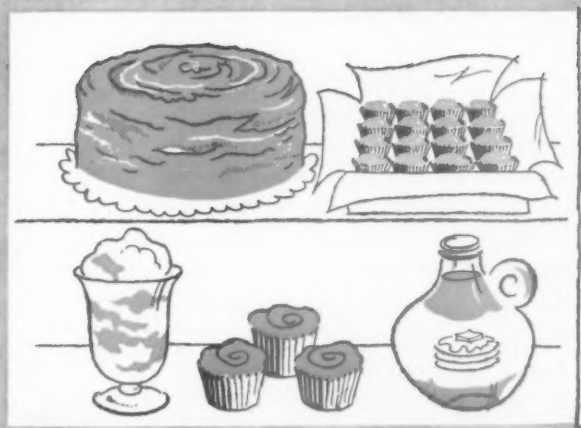

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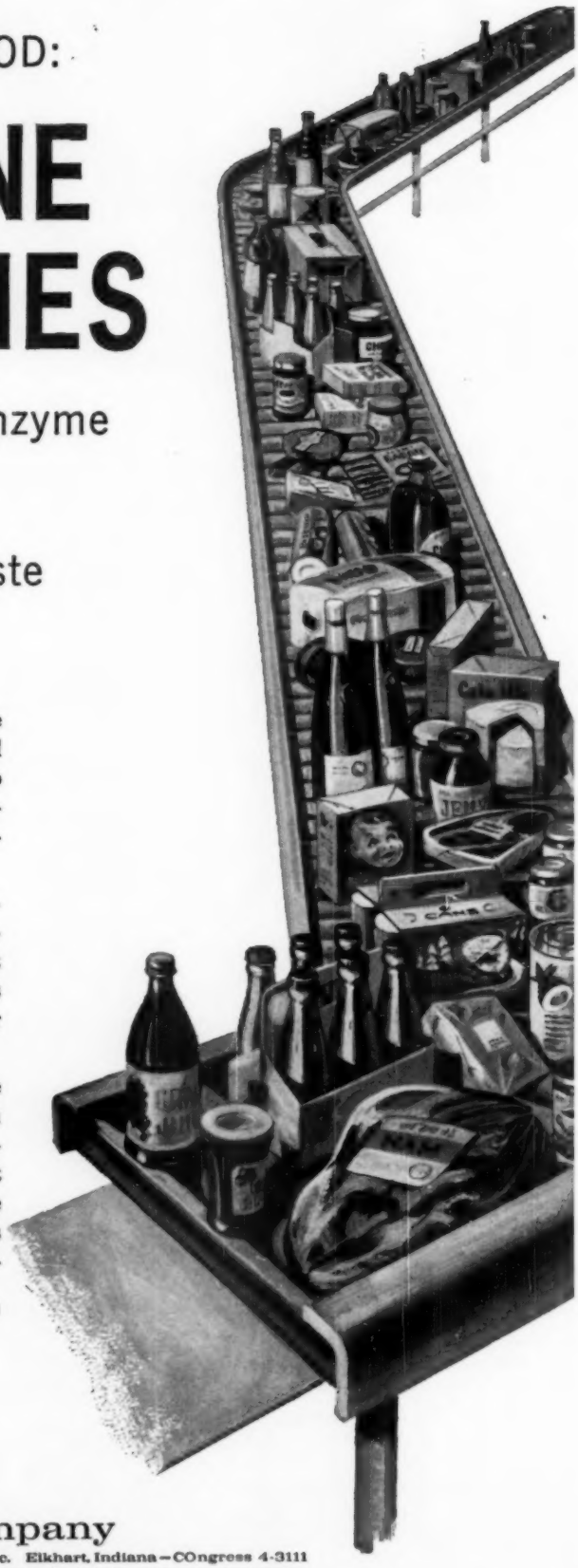

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Deterioration of Lemon Oil. Formation of *p*-Cymene from Gamma-Terpinene[a]

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(Manuscript received May 12, 1961)

SUMMARY

The formation of *p*-cymene from γ-terpinene during deterioration of lemon oil was shown. Citral, which was suspected as the precursor of *p*-cymene, was added to orange oil and allowed to deteriorate. No detectable amount of *p*-cymene was found. γ-Terpinene was isolated from lemon oil, and oxidized to *p*-cymene when exposed to air.

THE DETERIORATION of lemon oils and foods flavored with lemon oil is characterized by the development of a so-called "cymey" odor. Although *p*-cymene has long been suspected as causing this off-odor, its increase in deteriorated lemon oil has never been reported. Clark and Bernhard (1960) reported *p*-cymene in lemon oil, based on retention times on gas-liquid chromatography (GLC).

Citral, the principal flavoring constituent of lemon oil, has been accepted by many people as the precursor of *p*-cymene in deteriorated lemon oil. Price and Dickman (1948) isolated small quantities of *p*-cymene from an oil that separated from an aqueous reaction mixture of citral and phosphoric acid at 45°C. Strausz (1947) believed that *p*-cymene was formed from isocitral in lemon oil and that conversion was promoted by the presence of small amounts of fatty acids.

During an investigation of the composition of terpene hydrocarbons of lemon oils (Stanley *et al.*, 1961), *p*-cymene was found in several samples held in inadequately stoppered bottles. Some of these oils were completely colorless and had a strong "cymey" odor. Analyses of these oils are given in Table 1. It is apparent from these data that the appearance of *p*-cymene is paralleled by the disappearance of γ-terpinene. In fact, totals of these two components in deteriorated oils approach quite closely the average value for γ-terpinene obtained from samples of undeteriorated lemon oil. The average analyses of 105 samples of lemon oil held in well-stoppered bottles are shown in Table 1. Only trace amounts of *p*-cymene were found in these samples.

In more advanced examples of deterioration the *p*-cymene content of the oil ran between 6 and 8% of the terpene hydrocarbons (lemon oil contains approximately 85% terpene hydrocarbon). Citral, or citral alone, could not be the precursor of *p*-cymene found in the deteriorated lemon oils because citral itself seldom exceeds 3.5% of domestic oils (Stanley *et al.*, 1958).

In light of the above observation, further work was necessary in order to explain the formation of *p*-cymene in deteriorated lemon oil.

EXPERIMENTAL

Analysis of terpene hydrocarbons. Procedure for the chromatostrip-GLC analysis of the hydrocarbons of citrus oil is described in a previous paper (Stanley *et al.*, 1961). The method consists of spotting 0.04 ml of lemon oil on a chromatostrip and eluting with 2 ml of hexane. Eluate is analyzed directly by GLC.

Identification of p-cymene. In the hydrocarbon analysis of a deteriorated lemon oil, the vapor from the material having the retention time of *p*-cymene on GLC was bubbled into ethanol. The ultraviolet spectrum of this solution was found to be identical with the spectrum of known *p*-cymene.

p-Cymene was also identified by infrared analysis. Since a larger quantity of material was necessary for infrared analysis, a 30-ml sample of deteriorated lemon oil was deterpenated on silicic acid (Kirchner and Miller, 1952). Three hydrocarbon fractions were collected. Partial separation of the terpene hydrocarbons was observed in the three fractions. The second fraction contained the largest quantity of *p*-cymene. *p*-Cymene was isolated from this fraction by using a preparative scale GLC column (½ in. by 5 ft diethylene glycol succinate) and collecting the material having the retention time of *p*-cymene. Known *p*-cymene was purified similarly on a preparative scale GLC column. Infrared spectra of these two purified products were identical.

Experiments with citral. To check the possibility that citral gives rise to *p*-cymene, the following experiments were conducted.

A sample of citral that had been held at room temperature for at least 5 years without special precautions for excluding air, was analyzed for *p*-cymene by the chromatostrip-GLC method. No *p*-cymene was detected.

A 4% mixture of citral in orange oil was prepared by adding the required amount of a fresh sample of citral (citral content greater than 98% determined by vanillin-piperidine method) (Stanley *et al.*, 1958). The orange oil contained about 0.03%

Table 1. γ-Terpinene and *p*-cymene content of lemon oils.

	Per cent	
Undeteriorated oils	γ-Terpinene 10.3[a]	*p*-Cymene Traces
Deteriorated oils	8.7	1.5
	5.7	3.8
	2.9	7.4
	2.7	7.5
	1.4	7.4

[a] Average of 105 samples of lemon oil. Standard deviation of 0.93.

[a] Work supported in part by the Lemon Products Technical Committee, Los Angeles, Cal.

[b] Present address, see below.

[c] A laboratory of the Western Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture, 800 Buchanan Street, Albany 10, California.

citral and only a trace of γ-terpinene. The mixture was divided into two 50-ml portions. One portion was transferred to an empty glass-stoppered 250-ml Erlenmeyer flask and the other to a 500-ml glass-stoppered Erlenmeyer flask containing 200 ml of 6% aqueous citric acid solution. Flasks were stoppered to minimize evaporation losses of the lower-boiling components. The two flasks were held at room temperature and under usual lighting of the laboratory. Samples were removed periodically for analyses as described above. During this time the stoppers were removed from time to time to replenish air above the samples. After 3 weeks, during which time the color had completely bleached from the orange oil, no *p*-cymene was detectable. Analyses of the two samples, even after a period of 7 months, showed no detectable *p*-cymene.

Formation of p-cymene in lemon oil. Approximately 150 ml of lemon oil was placed in a 250-ml Erlenmeyer flask, loosely stoppered with glass wool, and allowed to stand at room temperature. *p*-Cymene and γ-terpinene content during 3 months were followed by the procedure described above. The results are plotted in Fig. 1.

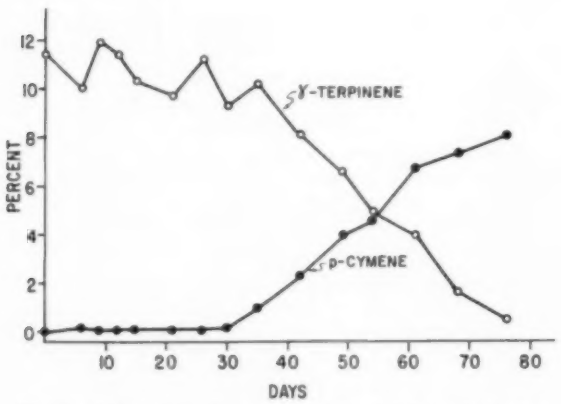

Fig. 1. Disappearance of γ-terpinene and formation of *p*-cymene during deterioration of a sample of lemon oil.

Formation of p-cymene from γ-terpinene. Pure γ-terpinene was isolated in the following manner. One pound of lemon oil was applied to a silicic acid chromatographic column, and the terpene hydrocarbons eluted with hexane (Kirchner and Miller, 1952). The terpene hydrocarbons were then fractionated in a spinning element column. γ-Terpinene fractions boiling at 60°C and 10 mm pressure were collected. The GLC analysis of the purest fraction showed over 99% γ-terpinene. The infrared spectrum of this fraction agreed with the spectrum of γ-terpinene reported by O'Connor and Goldblatt (1954).

A 7-ml sample of γ-terpinene was placed in a 50-ml Erlenmeyer flask, stoppered with a cotton plug, and allowed to stand at room temperature. Since hydrocarbon analysis by GLC gives only relative amounts of γ-terpinene and *p*-cymene and would not account for any non-volatile products, a spectrophotometric method was also used to determine the absolute amount of *p*-cymene. Points for the standard curve were determined by weighing known amounts of *p*-cymene in a volumetric flask and diluting with isooctane. Absorbance values used in the standard curve were the difference between the peak at 273 mμ and the trough at 271 mμ. This is to minimize error from other compounds that may absorb in this region. Fig. 2 shows the results of the oxidation of γ-terpinene to *p*-cymene as analyzed by GLC and by the spectrophotometric method.

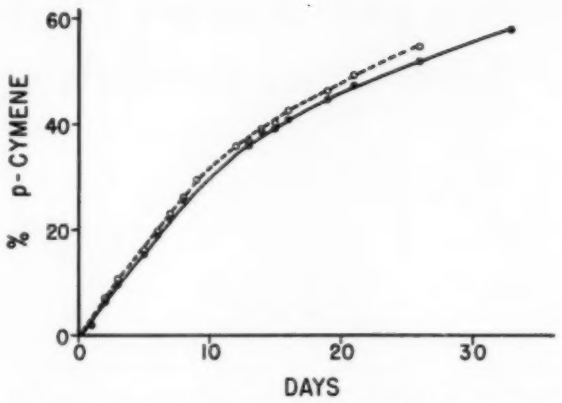

Fig. 2. Formation of *p*-cymene from a sample of γ-terpinene. Upper curve: analysis by spectrophotometer. Lower curve: analysis by GLC.

RESULTS AND DISCUSSION

From the evidence, it is definitely not possible for *p*-cymene in deteriorated lemon oil to have been derived from citral in any significant quantity. The amount of *p*-cymene in a badly deteriorated oil may exceed by several-fold the amount of citral originally present. Furthermore, no *p*-cymene could be detected when citral was added to orange oil. The citral added to the orange oil was identical to citral in lemon oil as analyzed by GLC. The principal path for the oxidation of γ-terpinene must be to *p*-cymene, since the disappearance of γ-terpinene can be accounted for by the appearance of *p*-cymene. Thus, γ-terpinene must be assumed to be the major precursor of *p*-cymene in deteriorated lemon oil, as shown by the experiments on the oxidation of γ-terpinene and of lemon oil.

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Hydrocarbon Composition of Lemon Oils and Its Relationship to Optical Rotation[a]

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(Manuscript received May 26, 1961)

SUMMARY

A simple, routine method of separating the terpene hydrocarbons from citrus oils for subsequent gas chromatographic analysis is described. Sabinene is identified in lemon oil, and α-thujene is tentatively identified by gas chromatographic retention time. Statistical results of the terpene hydrocarbon analyses of 105 lemon oil samples is reported. A direct relationship between *d*-limonene concentration and optical rotation and an inverse relation between β-pinene concentration and optical rotation were found.

ONE OF THE requirements in determining the purity of lemon oils is that their optical rotation must fall within certain arbitrary limits established by the Pharmacopeia of the United States (USP) (1955). According to these requirements the optical rotation must be between 57 and 65.6°. Optical rotation of lemon oils may vary from below 50 to above 70°.

No adequate explanation has been given for these wide variations in optical rotation. It is reasonable to assume that the terpene hydrocarbons ($C_{10}H_{16}$, hereinafter referred to as terpenes) have some relationship to optical rotation since they represent approximately 85% of the total composition of the oil. To resolve this question, the composition of the hydrocarbon fraction of lemon oils was studied.

The major terpenes have been isolated and identified both by the earlier classical techniques of distillation, preparation of derivatives, and chemical degradation (Guenther, 1949) and by the more recently developed gas-liquid chromatography (GLC) and comparison of retention times (Clark and Bernhard, 1960; Kung *et al.*, 1959; Liberti and Cartoni, 1958). In those studies octylene was identified tentatively and the following compounds identified positively, in lemon oil: α-pinene, β-pinene, camphene, *d*-limonene, β-phellandrene, and γ-terpinene.

Quantitative analysis of the individual terpenes was not possible in those earlier studies because methods then used involving distillation or conversion to solid derivatives were not adequate. The recently developed gas chromatographic methods of separation are sufficiently refined that quantitative analyses are possible. However, there is danger that components other than the hydrocarbons may interfere, and consequently it is desirable to make a preliminary separation. Separation of the hydrocarbons by column chromatography on silicic acid, although satisfactory for research studies (Kirchner and Miller, 1952), is not suitable for routine analyses.

To analyze many samples of oil on a routine basis, a more rapid method was developed for the separation and recovery of the terpenes based on chromatography on silicic-acid-coated chromatostrips (Kirchner *et al.*, 1951) using downward development (Stanley and Vannier, 1957) and elution. This method is particularly advantageous because the mixture eluted from the strip can be analyzed directly, i.e., without having to remove the eluting solvent.

EXPERIMENTAL

In analyzing the terpenes in citrus oils a 0.04-ml sample is applied to the chromatostrip with a calibrated micropipette, touching the tip of the pipette to the strip at a point 6½ in. from the bottom. The end of a wick of coarse filter paper is folded over the top of the strip and held in place with a small metal clip. The strip is then suspended in the side-arm test tube shown in Fig. 1 by a wire extending from the metal clip through the cork in the top of the test tube. The free end of the paper wick is drawn into the side-arm reservoir.

Development of the strip is started by adding about 5 ml of pure-grade *n*-heptane to the reservoir. Development of the strip is then allowed to proceed until 2 ml of eluate has collected in the bottom of the tube. The time required for development is about 4–5 hr. The strip is carefully removed, and the eluate is transferred to a small vial. The strip may be tested for completion of elution of the hydrocarbons by spraying with fluorescene-bromine reagent (Kirchner *et al.*, 1951). The eluate is analyzed directly by gas chromatography of a 50–100-μl sample.

Gas chromatographic analyses were made on an instrument equipped with a stainless-steel column packed with diethylene glycol succinate (DEGS) 10 ft by ¼ in. The column was operated at 80°C, 20 lb inlet pressure, and a helium flow rate of 120 ml per min. The terpene composition was calculated by the peak area as recorded by an integrator. Fig. 2 shows the chromatogram obtained for the terpenes of lemon oil.

Identification of the lemon oil terpenes was first made by comparison of retention times with known terpenes. For more conclusive evidence, the terpenes were isolated and their infrared spectra compared with those reported in the literature. For this purpose 1 lb of lemon oil was deterpenated on silicic acid as described by Kirchner and Miller (1952). The terpene fraction was fractionally distilled under vacuum in a spinning-element column. Since complete separation could not be made by vacuum distillation, the fractions were further purified by preparative-scale gas chromatography with a 5-ft by ½-in. DEGS column. Infrared spectra of these samples were determined and compared with those of known terpenes similarly purified. The infrared spectra were also compared with those reported by O'Connor and Goldblatt (1954). α-Pinene, β-pinene, myrcene, *d*-limonone, and γ-terpinene were identified by this procedure.

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[b] Present address: See below.

[c] A laboratory of the Western Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture, 800 Buchanan Street, Albany 10, California.

An unknown compound (I), Fig. 2, Peak 3, having retention time close to and just following β-pinene, was assumed to be sabinene, the major terpene hydrocarbon of oil of savin (Guenther, 1952). A sample of sabinene was obtained from oil of savin by vacuum fractional distillation followed by preparative scale GLC as described above. The identity of this material as sabinene was verified by its nuclear magnetic resonance (NMR) spectrum. The gas chromatographic retention time of sabinene from oil of savin was identical with that of compound (I) (peak 3). An enriched fraction containing 58.8% β-pinene and 41.2% of compound (I) (determined by gas chromatography) was recovered from lemon oil by silicic acid deterpenation, vacuum fractional distillation, and preparative-scale gas chromatography. Because the amount of material was limited, further purification was not attempted. This mixture was compared by infrared and NMR spectra with a known mixture of β-pinene and sabinene (gas chromatographic analysis indicated 59.3% β-pinene and 40.7% sabinene). The two mixtures were judged to be identical. The materials used for preparing the known mixture were pure sabinene from oil of savin and an enriched β-pinene fraction from lemon oil (91.5% β-pinene and 8.5% sabinene, determined by gas chromatography). The infrared spectra of the two mixtures and the infrared spectrum of sabinene appear in Fig. 3.

Presence of a second unknown terpene (II) in lemon oil was suspected because of the unsymmetrical shape of the α-pinene peak in the GLC analyses described above. This terpene, however, could be separated from α-pinene on a GLC column 10 ft by ⅛ in. packed with 2% DEGS on 80–100-mesh firebrick. Analyses were obtained with a hydrogen-flame ionization detector. Because of its retention time, this new peak, slightly behind α-pinene (1-a in Fig. 4), was assumed to be α-thujene. α-Thujene, reported to be present in oil of *Eucalyptus dives*, has a boiling point very close to that of α-pinene (Birch and Earl, 1938). The terpenes in a sample of oil of *Eucalyptus dives* were analyzed and found to contain 8% of a material having the same retention time as compound (II). A commercial fore-cut fraction (500 ml) from oil of *Eucalyptus dives*, a so-called α-phellandrene fraction containing 15% α-thujene, was fractionally distilled under vacuum in the rotating element column. The first 5 distillation fractions (*ca.* 100 ml) contained 75% α-thujene, 25% α-pinene, and traces of other terpenes. The second of these fractions was chromatographed on the preparative-scale GLC column, and a heart-cut in the α-thujene region was collected. The NMR spectrum of this material was correct for the structure of α-thujene. It had the same retention time on the 10 ft by ⅛ in. DEGS column as did compound (II) in lemon oil; therefore, it is assumed that compound (II) is α-thujene.

A linear response was demonstrated for both the tungsten filament and the hydrogen flame ionization detectors when a

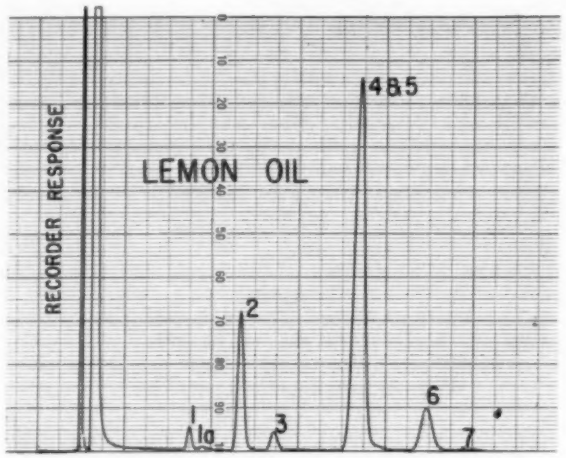


Fig. 2. Chromatogram of lemon oil terpenes. 80-μl sample, 85°C, 120 ml helium/min, thermal conductivity detector, 10-ft by ¼-in. column, 20% DEGS on 60–80-mesh firebrick. Peaks identified in Table 2. Length of time for complete chromatograph approx 15 min.

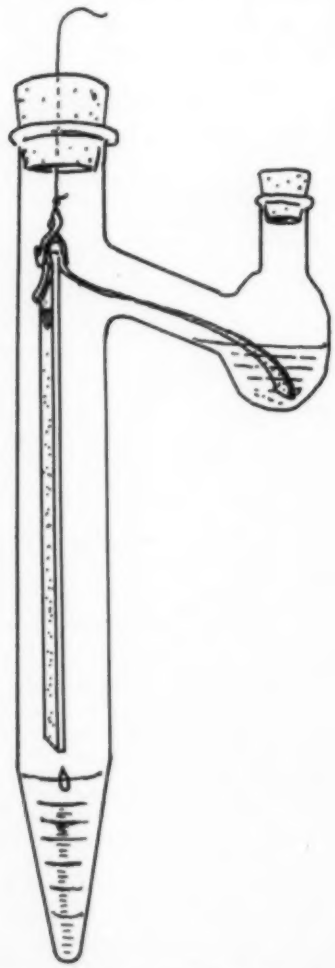


Fig. 1. Apparatus for separating terpenes on chromatostrips.

Table 1. Response and recovery experiment for a terpene mixture of known composition.

Terpene	Calcd. comp. (wt %)	Gas chromatographic analyses (area %)		
		Original mixture		After elution from silicic acid chromatostrip (¼ in DEGS)
		¼ in DEGS filament	⅛ in DEGS flame	
α-Pinene	9.3	9.2	9.2	9.2
		9.2		9.6
		8.9		9.4
β-Pinene	14.6	14.3	14.3	12.8
		14.0		12.3
		13.9		13.4
d-Limonene	64.3	64.3	63.1	66.1
		65.1		66.4
		65.2		65.0
γ-Terpinene	11.8	12.2	12.3	11.9
		11.6		11.6
		12.0		12.1

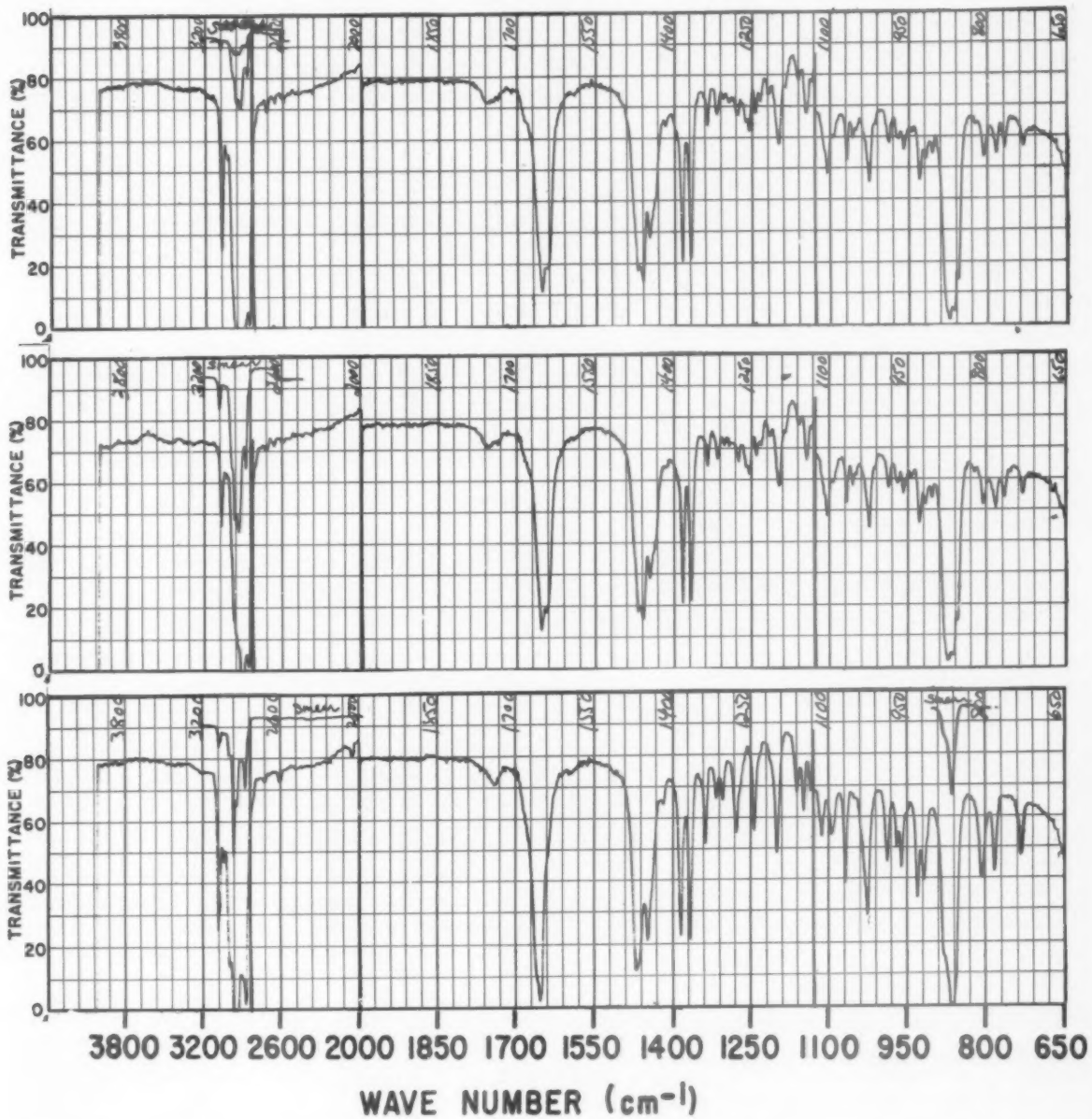


Fig. 3. Infrared spectra of mixtures of β-pinene and sabinene, and the spectrum of sabinene. Top: β-pinene and sabinene mixture isolated from lemon oil. Middle: known mixture of β-pinene and sabinene. Bottom: sabinene isolated from oil of savin.

terpene mixture of known composition was used. The mixture was prepared with weighed amounts of terpenes purified by vacuum distillation and preparative-scale GLC. The data for the calculated composition and the direct GLC analyses of the original mixture appear in Table 1. It is evident that the response characteristics of the two detector systems are satisfactory for the analysis of the terpenes.

To show that no isomerization of the terpenes occurs on silicic acid and to check recoveries during the chromatostrip determination, 0.04 ml of a mixture of terpenes of known composition was applied to a silicic acid chromatostrip and eluted with *n*-heptane according to the analytical method described above. These results are tabulated in Table 1. Results indicate that there is no significant isomerization of the terpenes or change in composition by this method of analysis. The apparent stability of the terpenes on chromatostrips confirms a recent report that isomerization of the terpenes on silicic acid occurs only when the silicic acid is contaminated with aluminum oxide (Rudakov and Shestaeva, 1959). This is contrary to an earlier statement that these terpenes were isomerized on silicic acid (Arbuzov and Isaeva, 1953).

Representative samples of commercial lime, grapefruit, and California orange oils were analyzed for hydrocarbons by the standardized procedure. The respective chromatograms appear in Figs. 5, 6, and 7 (for comparison with the lemon oil chromatogram in Fig. 2). Similarities in the distribution patterns of the terpenes parallel those observed for the aldehydes (Stanley *et al.*, 1961). Lemon oil resembles lime oil; orange oil resembles grapefruit oil.

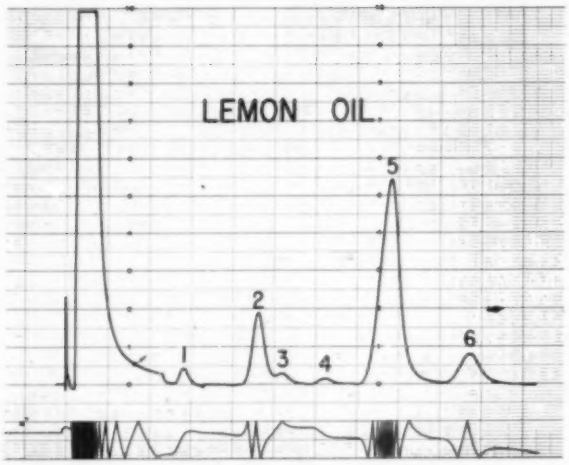


Fig. 4. Chromatogram of lemon oil terpenes. 1-μl sample, 70°C, 10 ml N_2/min, flame ionization detector, 10-ft by ⅛-in. column, 2% DEGS on 80–100-mesh firebrick. Peaks identified in Table 2, peak 1-a α-thujene.

RESULTS AND DISCUSSION

A series of 105 samples of lemon oil covering over 5 years of production from a local citrus processor were analyzed by the method described. Results are summarized in Table 2.

Table 2. Results from the terpene hydrocarbon analysis of 105 samples of lemon oil.

Terpene	Peak no.	Per cent	
		Average	Standard deviation
α-Pinene	1	2.46	0.32
β-Pinene	2	14.8	3.25
Sabinene	3	1.67	0.56
Myrcene	4	1.10	0.38
d-Limonene	5	69.7	4.50
γ-Terpinene	6	10.3	0.93
p-Cymene	7	Trace	

Wide variations in the optical rotation of lemon oils have been observed, and these variations are found to be greatest between oils from coastal and inland areas. The rotation of inland oils may run as high as 70°, whereas that of coastal oils may fall below 50°. There are also pronounced seasonal variations within each growing area. The suggestion has often been made that the changes in optical rotation are caused by the presence of *l*-limonene as well as *d*-limonene. However, examination of the optical rotation in relation to the terpene data indicated that the wide variations in optical rotation were due to variations in relative amounts of β-pinene and *d*-limonene. *d*-Limonene and *l*-β-pinene have $[\alpha]^{20}_{D}$ of +126.6 and −23.4, respectively (Pliva and Herout, 1950). γ-Terpinene is optically inactive, and the other terpenes are present in such small quantities that they do not exert any significant influence on rotation.

Fig. 8 plots *d*-limonene concentration against the optical rotation for the oil samples analyzed in this study. Although there is considerable scattering in the data, the trend is quite obvious. β-Pinene content is plotted against the optical rotation in Fig. 9. The relationship of the optical rotation to β-pinene is more consistent than that of *d*-limonene. The effect of the other minor constituents has not been considered in these graphs. It is reasonable to asume that the variation in β-pinene content contributed heavily to the observed changes in the optical rotation of lemon oil. In contrast to lemon oil, orange and grapefruit oils have optical rotations close to 100°. Over 95% of the terpenes of these latter oils is *d*-limonene.

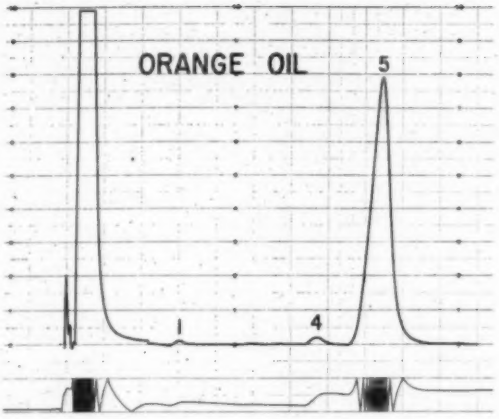


Fig. 5. Chromatogram of orange oil terpenes. 80-μl sample, 82°C, 120 ml helium/min, thermal conductivity detector, 10-ft by ¼-in. column, 20% DEGS on 60–80-mesh firebrick. Peaks identified in Table 2. Length of time for complete chromatograph approx 15 min.

Five samples of foreign lemon oils were analyzed in order to determine whether these relationships between optical rotation and terpene composition were universally applicable. The results (solid circles in Figs. 8, 9) followed the same pattern observed for domestic lemon oils.

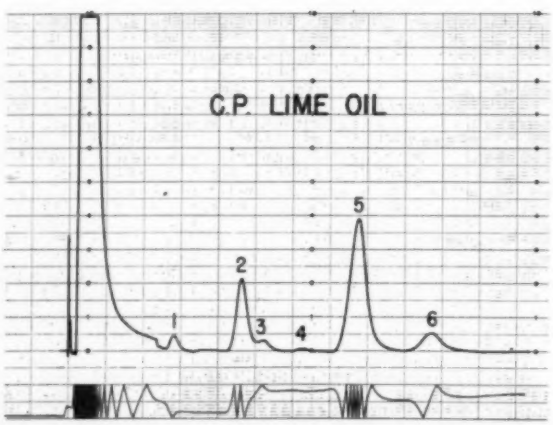
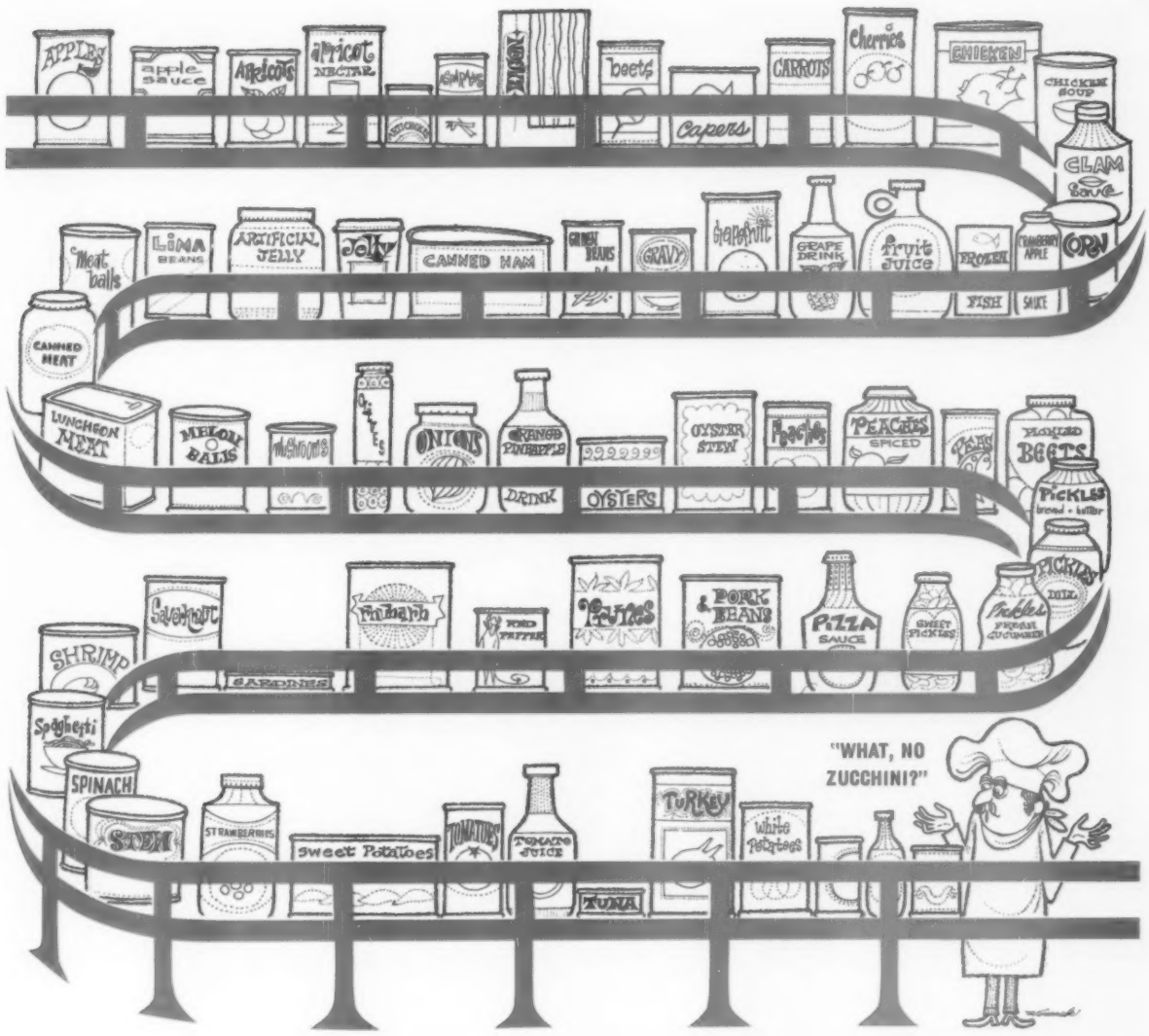


Fig. 6. Chromatogram of C.P. lime oil terpenes. 80-μl sample, 85°C, 120 ml helium/min, thermal conductivity detector, 10-ft by ¼-in. column, 20% DEGS on 60–80-mesh firebrick. Peaks identified in Table 2. Length of time for complete chromatograph approx 15 min.



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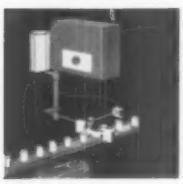

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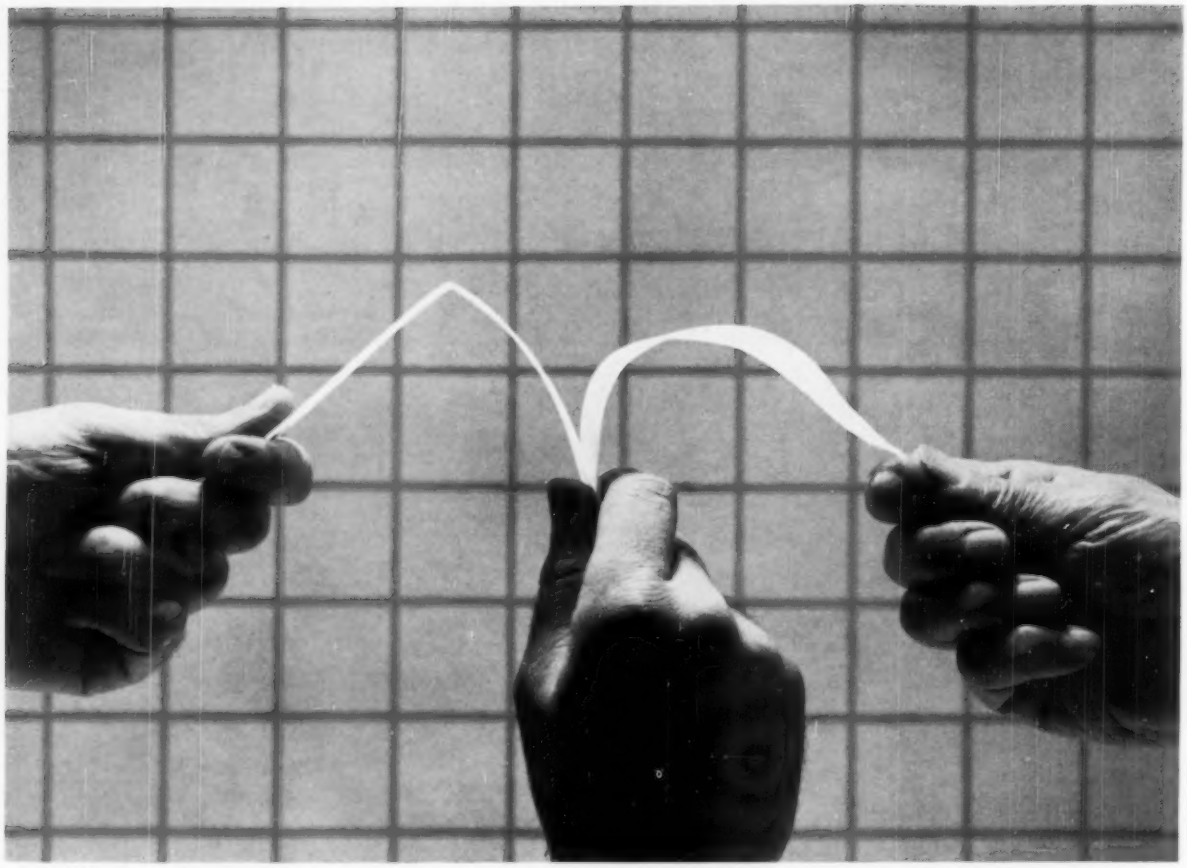

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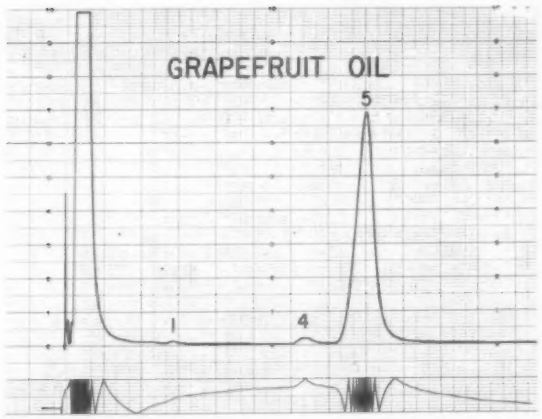

Fig. 7. Chromatogram of grapefruit oil terpenes. 80-μl sample, 85°C, 120 ml helium/min, thermal conductivity detector, 10-ft by ¼-in. column, 20% DEGS on 60–80-mesh firebrick. Peaks identified in Table 2. Length of time for complete chromatograph approx 15 min.

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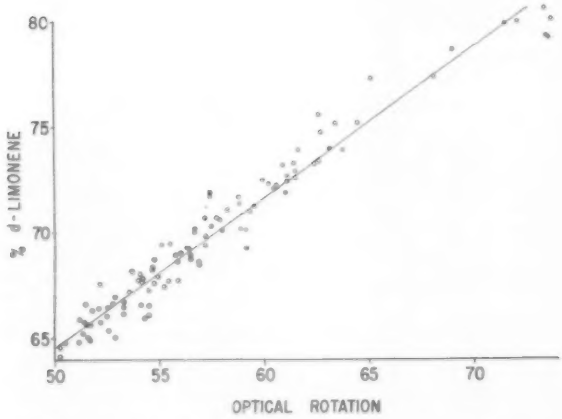

Fig. 8. Plot of per cent *d*-limonene against optical rotation; ○—domestic samples, ●—foreign samples.

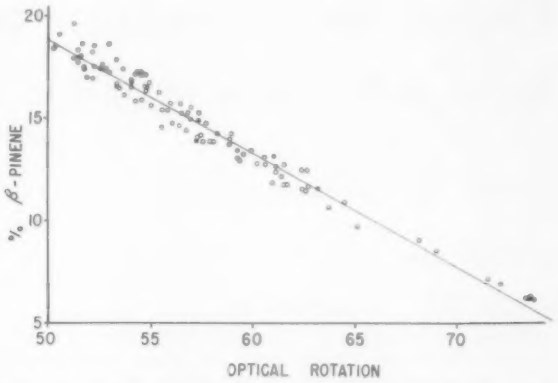

Fig. 9. Plot of per cent β-pinene against optical rotation; ○—domestic samples, ●—foreign samples.

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Effect of Ionizing Radiation on Cakes and Biscuits Made with Milled Irradiated Wheat *

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SUMMARY

Wheat was treated with ionizing radiation at 23,000, 46,000, 70,000 and 93,000 rad. The treated, along with a sample of untreated, wheat was milled into cake flour and all-purpose flour. Plain cakes and biscuits were respectively prepared with cake flour and all-purpose flour to determine the effect of radiation on the quality of the finished products. Untreated wheat flour was used to produce a standard product.

Panel scores and physical measures of plain cake showed no differences in the baking quality of flours made from wheat treated with as much as 93,000 rad ionizing radiation. Panel scores for biscuits made with irradiated milled wheat showed significant differences in all attributes tested: color of crust and crumb, flavor, tenderness, texture, and acceptability. The 23,000 and 70,000 rad samples showed no differences from the standard except that the 70,000 rad sample was scored less desirable in flavor. The 46,000 rad product was scored lower than the standard in flavor, tenderness, and texture, and the 93,000 rad product scored lower than the standard on all attributes except texture. The physical measures on the biscuits indicated only that the 70,000 rad and standard doughs were slightly more compressible and softer than the others, but there were no differences in dough elasticity or biscuit volume.

THE USE of ionizing radiation has been proposed to control insect infestation of grain during storage. Hassett and Jenkins (1952) reported that 25,000 rep are sufficient to control insect infestation. Nicholas and Wiant (1959) found lethal doses of cathode-ray radiation to be 40,000–60,000 rep for moths and 10,000–20,000 rep for beetles. Some studies have been made on the effects of radiation at these levels and above on the baking quality of flours and on the palatability of baked products. Group threshold for detection of off-flavors, established by the probit, lies at about 50,000 rep in yeast breads made with milled irradiated wheat and with irradiated flour (Nicholas *et al.*, 1958). Milner and Yen (1956) reported that commercial-type yeast bread made with milled irradiated wheat showed improvement in volume and loaf scores at 125,000-rep treatment. Cakes made with bread flour irradiated by 100,000 rep exhibited a sweeter flavor than the control (Brownell *et al.*, 1955). Cakes made with all-purpose, cake, and bread flours given 20,000 rep showed no differences from the control. Biscuits made with the irradiated bread flour at levels greater than 20,000 rep were gummy, and off-flavors were noticed. Alsup (1959) reported that, with the triangle difference test, radiation flavor was detectable in biscuits made with flour irradiated at 50,000 rep. Flavor, tenderness, and texture scores, however, were not significantly different from the control for biscuits made with flour irradiated at 100,000 rep or less.

Only two of these studies (Milner and Yen, 1956; Nicholas *et al.*, 1958), reported irradiation of the wheat before milling. Since grain would be irradiated before storage to prevent losses from insect infestation and since flour would rarely be irradiated under practical conditions, the studies reported herein were made on flour milled from irradiated wheat. The aim of this study was to determine the effect of radiation, at levels lethal to insects in the grain, on the baking quality of biscuits made from all-purpose flour and plain cake made from cake flour. Previous reports on baked products have indicated that the product made from irradiated flour was scored less desirable if panel members were informed of the radiation treatment (Alsup, 1959; Nicholas *et al.*, 1958). It was thought that if the panel members were told only that there was a slight variation in ingredients, the possibility of prejudice against radiation would be removed from the judging.

EXPERIMENTAL PROCEDURE

A commercial milling company, International Milling Co., Minneapolis, Minnesota, supplied 150 lb of wheat for all-purpose flour and 100 lb for cake flour. Part of the wheat was treated with ionizing radiation from a one-million-electron-volt generator of the resonant transformer type at levels of 23,000, 46,000, 70,000, and 93,000 rad (the absorption of 100 ergs per gram of material). The irradiated wheat, along with the remaining untreated wheat, for control purposes, was shipped to the commercial company for milling. A Beta Chlora bleach was used in the cake flour. The all-purpose flour was not bleached.

Cakes were prepared according to the formula in Table 1. Flour from each radiation level was tested with every other radiation level twice, resulting in 6 tests of each radiation

Table 1. Formula and method for plain cake.

Ingredients	Measure
Hydrogenated shortening	56 g
Sugar	150 g
Egg	48 g
Evaporated milk	59 cc
Distilled water	59 cc
Cake flour	150 g
Baking powder	5 g
Salt	1 g

Method: Cream fat and sugar. Add beaten egg. Sift flour, baking powder, and salt. Add to creamed mixture. Add milk and water. Scale 215 g batter into each of two 5 × 2½ × 2-in. pans lined with waxed paper. Bake 36 minutes in 360°F oven.

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level. One recipe of cake from the control flour and two from test flours were prepared for each of 12 testing days.

Six trained tasters used a multiple-comparison scoring system for testing. They were asked to compare each unknown sample with the identified control sample as to color, flavor, tenderness, texture, and acceptability. Each attribute was rated: 5, better than the standard; 4, equal to the standard; 3, slightly below standard; 2, definitely below standard; or 1, extremely below standard. Panel members were not told that the flours were irradiated.

Biscuits were prepared from the all-purpose flour according to the formula in Table 2. Biscuits were prepared from control flour and the four irradiated flours on each of 6 testing days. Six trained judges scored the biscuits as described. Again, they were not told that the flours were irradiated.

Table 2. Formula for biscuits.

Ingredients	Measure
All-purpose flour	300 g
Baking powder (SAS)	10 g
Salt	2 g
Hydrogenated shortening	50 g
Milk	215 cc

Volume and compressibility of the baked cakes were measured. Compressibility and elasticity of the dough, and volume of the baked product, were recorded for the biscuits. A standard loaf volume meter that measures directly in cc, and a micrometer adjustment penetrometer measuring in 1/10 mm, were respectively used to determine volume and compressibility-elasticity. One loaf cake and five biscuits were used for volume measurements. Compressibility was recorded in 1/10 mm as the reduction of height of a core of 1 in. diameter and ½-in. height caused by a weight of 72.6 g resting on the surface for 15 sec. Elasticity of the biscuit dough was recorded in 1/10 mm as the change in height 15 seconds after the weight had been removed.

DISCUSSION AND RESULTS

Table 3 gives panel scores for exterior and interior color, flavor, tenderness, texture, and over-all acceptability for the cakes. An analysis of variance showed no differences for any of the attributes.

Table 3. Mean panel scores for cakes made with milled irradiated wheat.

	Level of radiation (rad)				
	Standard	23,000	46,000	70,000	93,000
Exterior color	4.0	4.0	4.0	4.0	4.1
Interior color	4.0	4.0	4.0	3.8	3.9
Flavor	4.0	4.0	3.9	3.8	3.8
Tenderness	4.0	4.0	3.9	3.8	3.9
Texture	4.0	3.9	4.0	3.8	3.7
Acceptability	4.0	4.0	3.9	3.8	3.8

Analyses of variance indicated that volume and compressibility of the cakes were not affected (Table 4). Thus, plain cakes made from wheat treated with ionizing radiation at levels lethal to insects appear to be acceptable. It will be noted, however, that in all attributes other than exterior color there was a trend for panel scores to be slightly lower for cakes from wheat treated with 46,000, 70,000, and 93,000 rad.

Table 4. Mean volume and compressibility values for cakes made with milled irradiated wheat.

	Level of radiation (rad)				
	Standard	23,000	46,000	70,000	93,000
Volume (cc)	2,790	2,792	2,795	2,801	2,794
Compressibility (1/10 mm)	21.4	21.3	21.5	22.1	20.9

Analyses of variance of panel scores for biscuits showed significant differences for each attribute tested. Flavor and acceptability showed highly significant differences at the 1% level of probability; the others were significant at the 5% level. Table 5 shows the mean panel scores for six judges, who scored each treatment six times. The mean panel scores indicate that the biscuits from treated wheat were equal to the standard product or less desirable, but never superior in any attribute tested. The analyses of variance of panel scores showed that wheat irradiated at 23,000 rad before milling produced biscuits like the standard biscuit in all attributes; with 46,000 rad, the biscuits were different from the standard in flavor, tenderness, and texture; with 70,000 rad, flavor was less desirable than the standard; and with 93,000 rad, crust and crumb color, flavor, and tenderness were less desirable than in the standard product. Although these differences were recorded by the panel, only the biscuits from the 93,000 rad treatment were scored less acceptable than the standard product. There is no ready explanation for the tendency of the panel to score 70,000 rad biscuits higher than 46,000 rad biscuits in most attributes.

Table 5. Mean panel score for biscuits made with milled irradiated wheat.

	Standard	23,000	46,000	70,000	93,000
Crust color	4.0	3.8	3.9	3.9	3.6[a]
Crumb color	4.0	4.0	4.0	3.9	3.8[b]
Flavor	4.0[c]	3.9[c]	3.5	3.7	3.7
Tenderness	4.0	3.9	3.7[d]	3.9	3.7[d]
Texture	4.0	4.0	3.6[e]	3.8	3.8
Acceptability	4.0	3.8	3.7	3.8	3.6[f]

[a] Significantly lower than the standard and 46,000- and 70,000-rad biscuits.
[b] Significantly lower than the standard and 23,000- and 46,000-rad biscuits.
[c] Significantly higher than the 46,000-, 70,000-, and 93,000-rad biscuits.
[d] Significantly lower than the standard biscuits.
[e] Significantly lower than the standard and 23,000-rad biscuits.
[f] Significantly lower than the standard biscuits.

At the levels used, radiation resulted in no differences in biscuit volume or dough elasticity as tested by analyses of variance. However, the doughs from the standard and 70,000 rad treated flours were significantly more compressible and softer than the dough from the 93,000 rad flour (Table 6).

Thus the panel was able to identify differences in biscuits at the various levels of radiation treatment, yet they recorded cake scores that indicated no differ-

Table 6. Mean volume, compressibility, and elasticity values of biscuits made with milled irradiated wheat.

	Standard	23,000	46,000	70,000	93,000
Volume (cc)	2,491	2,492	2,497	2,494	2,491
Compressibility (1/10 mm)	67.3[a]	60.6	60.8	65.9[a]	57.1
Elasticity (1/10 mm)	25.7	24.7	29.3	34.0	29.0

[a] Doughs significantly more compressible than doughs from 93,000-rad-treated wheat flour.

ences at the same levels of radiation. It may be that the sugar, egg, and/or milk in a cake formula or the bleach used in the cake flour helped to mask the changes due to radiation. More likely, however, the amount of flour present in the two products was the determining factor in the differences evidenced. The plain cake contained only 28% flour, whereas the biscuits were 52% flour. With flour concentration nearly twice as great in the biscuits as in the cake, more differences would be expected to appear. The biscuit formula used was very lean. A richer formula, more fat in relation to flour, might produce a biscuit more like the standard product. It should be remembered that only 93,000 rad resulted in a product less acceptable than the standard, and that this radiation level is higher than that considered necessary for insect control.

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The Determination of Soluble Solids in Citrus Juices. III.

Empirical Factors for Converting Refractometer and Density Values to Soluble Solids

(Manuscript received December 12, 1960)

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SUMMARY

The soluble solids contents, as determined by vacuum drying, of a series of concentrated orange and grapefruit juices and single-strength orange and grapefruit juices were compared with their sugar scale values as determined by refractometer, hydrometer, and pycnometer. The average ratios of soluble solids to observed sugar scale values were:

	Refractometer	Hydrometer	Pycnometer
Single-strength orange juice	**0.995**	**0.970**	**0.957**
Concentrated orange juice	**.979**	.....	**.969**
Single-strength grapefruit juice	**.992**	**.969**	**.961**
Concentrated grapefruit juice	**.996**	.....	**.987**

These ratios are suggested for use as factors for converting observed sugar scale values to percent soluble solids for improved accuracy without loss of precision.

[a] One of the laboratories of the Southern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture.

THE FIRST PAPER of this series (Scott *et al.*, 1960) showed that there are besides citric acid at least seven nonsucrose components that significantly affect the accuracy of refractometer sugar scale values for citrus juices. The second paper (Scott *et al.*, 1961) reported analyses for these components and corrections applied for each to the refractometer values of a series of concentrated orange and grapefruit juices. The multiple correction factors, on the average, considerably improved the correlation of refractometer values with soluble solids. In this study the possibility of developing a simple empirical factor for converting refractometer values to soluble solids was explored as an alternate to the multiple correction method. Likewise, factors for converting hydrometer and pycnometer Brix values to soluble solids were investigated.

EXPERIMENTAL

Selection of samples. Fourteen samples of concentrated orange juice (12 of 4-fold and 2 high density) were obtained

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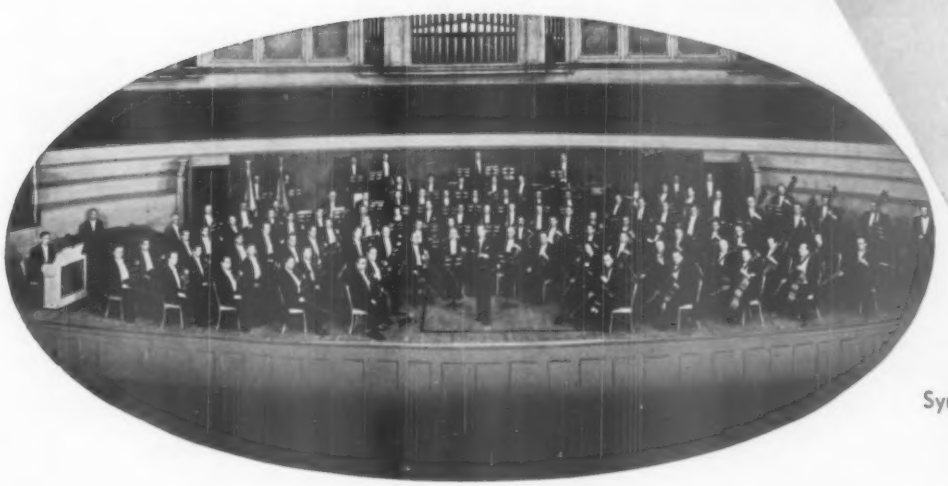

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either in retail packages or from processing lines of commercial plants in January and February, 1959. Twelve samples of concentrated grapefruit juice were obtained, 8 in retail packages and 4 from laboratory packs prepared for other purposes. Since analyses for soluble solids by drying and refractometer values were available from the studies reported in the previous paper for 32 samples of concentrated orange juice (23 of 4-fold and 9 high density) and 8 samples of concentrated grapefruit juice, those results were included in this study.

For the study of single-strength juices, 4 samples of fresh orange juice were obtained from processing plants, 7 samples of canned orange juice were obtained either from plants or from laboratory packs, and 4 samples of grapefruit juice were obtained from canneries. In addition, the 14 samples of concentrated orange juice and 12 of concentrated grapefruit juice were diluted and treated as single-strength juices. Consequently, data are presented on 25 orange juices and 16 grapefruit juices of approximately single strength.

Soluble solids. Separation of suspended matter, or insolubles, from concentrated juices required their dilution. Samples of about 175 g were weighed on an analytical balance, diluted at 20°C to 700 ± 0.3 ml, and analyzed similarly to single-strength juices, as follows. Fifty-ml portions were centrifuged at 1600 r.p.m. with a tip radius of 10 in. for 20 min in capped tubes, the temperature again adjusted to 20°C, and 15.0-ml portions of the supernatant were pipetted onto sand in tared weighing dishes. Duplicate dishes were used in each instance, with volumetric samples being taken from separate centrifuge tubes. Visible moisture was removed from the dishes on a steam bath, and drying was completed in a vacuum oven at 60°C under 29 in. of vacuum for 30 hours.

To check the efficiency of the above method in removing insoluble solids, a sample of commercial frozen concentrated orange juice was reconstituted and 1200 ml was centrifuged as above. All supernatants were combined. Solids were determined on one portion of the supernatant, as above. A second portion was further centrifuged for one hour, and the resulting supernatant dried in the same manner. A third portion was filtered with diatomaceous filter-aid and suction, and the filtrate similarly dried. Determinations of dry solids were made on each of the three clarified portions in quintuplicate, using the same pipette to measure all aliquots into the weighing dishes, and all 15 dishes were dried simultaneously. All replicates except one agreed within 0.04% solids. Centrifuging for the additional hour reduced average solids content by only 0.02% solids, whereas filtering reduced solids content from that of the original supernatant by 0.10%. Since diatomaceous filter-aid is known to adsorb compounds of high molecular weight and/or electrically charged colloids, it is thought that part of the 0.1% reduction in solids by filtering was the result of the removal of pectin. The measurement of soluble solids by weighing those solids remaining after centrifuging for 20 minutes is therefore considered sufficiently accurate for the purposes of this study.

Total solids. Single-strength juices or reconstituted concentrates were pipetted, without centrifuging, onto sand in tared weighing dishes and dried as for soluble solids.

Refractometer values. Refractometer values were determined on all single-strength or reconstituted juices before and after centrifuging, and on all concentrates. Readings were taken with a Bausch and Lomb Precision Refractometer adjusted to 20 ± .05°C with circulating water. (The mention of trade products does not imply that they are recommended by the Department of Agriculture over similar products not mentioned.) Calibration of the instrument was checked periodically with 2,2,4-trimethylpentane, certified by the National Bureau of Standards, and distilled water. Room temperature did not exceed that of the instrument by more than 5°C at any time. Since the scale of the instrument is calibrated in arbitrary units, scale readings were converted to equivalent sucrose percentages (sugar scale values) with tables provided by the manufacturer.

Hydrometer Brix. Hydrometer Brix values were taken on all single-strength and reconstituted juices both before and after centrifuging. Two hydrometers, with scales 0–11° and 9–21°, were calibrated against a National Bureau of Standards certified hydrometer. Readings were taken at 20.0°C; juice, hydrometer, and cylinder were all precooled to the standard temperature. The 0–11° hydrometer was used only when Brix values were less than 10°. The hydrometers were graduated to 0.1°, and readings were estimated only to the closest 0.05°.

Pycnometer Brix. Pycnometer Brix values were determined on single-strength and reconstituted juices both before and after centrifuging. Specific gravity of samples was taken by weighing to the 4th decimal place in 25-ml specific-gravity bottles with thermometers and capped sidearms. Temperatures of both juice and bottles were adjusted to 20°C before filling, and weighings were made within 5 minutes of filling. The weight of concentrate or juice was compared to the weight of water at the same temperature. Specific gravities were converted to degrees Brix with Table 109, National Bureau of Standards Circular 440.

RESULTS AND DISCUSSION

Solids determinations by each method in single-strength juices, 24 of orange and 16 of grapefruit before and after centrifuging, were averaged (Table 1).

Table 1. Comparison of per cent solids in whole and centrifuged juices as determined by various methods.

	Average values					
	24 orange juices			16 grapefruit juices		
	Whole	Cent.	Diff.	Whole	Cent.	Diff.
Vacuum drying	11.628	11.415	0.213	10.242	10.175	0.067
Refractometer	11.436	11.344	.092	10.265	10.252	.013
Hydrometer	11.723	11.525	.198	10.552	10.445	.107
Pycnometer	11.883	11.593	.290	10.585	10.517	.068
Titratable acid	.798	.785	.013	1.185	1.182	.003

The removal of suspended matter from citrus juices by centrifuging is possible because of its high density. D. A. Morgan, formerly of this Laboratory, found the density of washed and dried pulp to be about $1.75^{20/4}$. The removal of this heavy material, of course, changes the relative composition of juice. In three of the methods, vacuum drying, hydrometer Brix, and pycnometer Brix, the weight of the suspended matter is measured along with the dissolved solids. Consequently, differences due to centrifuging are substantial. The two values of solids by vacuum drying for each juice actually indicate total solids and soluble solids by definition.

Refractometer values should be the same for whole and centrifuged juices. The differences shown are small, and may be within experimental error. Differences between percentages by weight of titratable acid were also probably within experimental error.

The insoluble solids content of grapefruit juice is usually lower than that of orange juice. Consequently, differences between whole and centrifuged juices were less for grapefruit than for orange.

To compare the compositions of concentrated or single-strength juices of varying concentration on a common basis, ratios were calculated between soluble solids by drying and degrees Brix as determined by each of the other methods. These ratios serve as factors by which observed values can be converted to soluble solids.

Table 2 illustrates derivation of the factors for converting refractometer values and pycnometer values of concentrated orange juice to soluble solids. To con-

Table 2. Derivation of factors for converting refractometer values and pycnometer values of concentrated orange juice to soluble solids.

Soluble solids (A)	Refrac. values (B)	Pycn. values (C)	Factors for converting to soluble solids: Refrac. (A/B)	Factors for converting to soluble solids: Pycnom. (A/C)
42.85	43.69		0.981	
40.68	41.73		.975	
40.60	41.79		.972	
40.63	41.41		.981	
40.09	41.36		.969	
40.73	41.57		.980	
40.65	41.69		.975	
40.64	41.50		.979	
40.20	41.60		.966	
40.14	41.47		.968	
40.26	41.42		.972	
40.60	41.68		.974	
40.18	41.69		.964	
40.47	41.69		.971	
40.53	41.49		.977	
40.70	41.55		.980	
41.42	42.03		.986	
40.83	41.53		.983	
40.84	41.78		.978	
40.80	41.41		.985	
40.08	41.25		.972	
40.38	41.04		.984	
41.35	41.55	41.76	.995	0.990
43.18	44.26	45.06	.976	.958
43.18	44.42	45.14	.972	.957
43.03	43.40	43.94	.992	.979
43.57	44.50	45.22	.979	.963
40.52	41.49	42.16	.977	.961
43.91	44.62	45.37	.984	.968
40.54	41.04	41.55	.988	.976
40.60	41.09	41.70	.988	.974
40.91	41.33		.990	
44.79	45.36	46.33	.987	.967
43.29	44.26		.978	
39.78	41.03		.970	
Average			.979	.969
Maximum deviation from average			.016	.031
Standard deviation			.007	.010

serve space, only values of 4-fold concentrates are shown. Inclusion of the 11 higher-density concentrates would not change the derived results. The average soluble solids/refractometer ratio for the 35 samples was 0.979, with a maximum variation from this average of 0.016 and a standard deviation of 0.007. The average soluble solids/pycnometer Brix ratio for the 10 samples analyzed was 0.969, with a maximum variation from the average of 0.021 and a standard deviation of 0.010.

Similar factors were developed for converting observed Brix values to soluble solids for single-strength orange juice and concentrated and single-strength grapefruit juice. Individual values are not shown, but derived results are summarized in Table 3. The number of samples of each type of juice from which the averages were drawn is shown in parentheses following the method. For single-strength orange juice the average factor for converting refractometer Brix to soluble solids was 0.995, with a standard deviation of 0.017. Maximum observed deviation from the average among the 24 samples was −0.038. Application of this factor to juice having a refractometer Brix value of 12.00° would indicate it to contain 11.94% soluble solids (0.995×12). Among the samples examined, the standard deviation of results of this average soluble solids content was 0.20° Brix, with a maximum deviation of −0.46% solids. Statistical analysis indicates that among similar samples examined by similar methods, 19 of 20 should deviate from the 0.995 factor by less than 0.044, or ±0.53% solids for 12.00° Brix juices.

Similar analyses of the ratios of soluble solids to hydrometer Brix and to pycnometer Brix of single-strength juices show that such ratios are lower, and that deviations from the average are of about the same order of magnitude as deviations from the per cent soluble solids/refractometer Brix ratio.

Since hydrometers could not be used with concentrated juices, only the relationships of soluble solids to refractometer Brix and to pycnometer Brix could be determined. For 46 samples of concentrated orange juice, the ratio of soluble solids to refractometer Brix was the same as reported above for 35 samples of 4-fold concentrate, 0.979:1. According to these data, concentrated orange juices having refractometer values of 42.00° Brix would contain an average of 41.12% soluble solids. The largest observed variation from this average was +0.67, or 41.79%.

Observed refractometer values were closer to solu-

Table 3. Ratios of soluble solids to degrees Brix as determined by refractometer, hydrometer, and pycnometer, with standard deviations and maximum deviations from average ratios, and examples.

Method and number of samples	Average ratio	Deviation from average ratio: Std.	Deviation from average ratio: Max.	Per cent soluble solids at: 12° Brix	Per cent soluble solids at: 42° Brix	Confidence[b]: Dev. from average: Ratio	Confidence[b]: Dev. from average: Sol. solids
Single-strength orange juice							
Refrac. (24)	0.995	0.017	−0.038	11.94 ± .20(−.46)[a]		.044	0.53%
Hydr. (24)	.970	.016	− .039	11.64 ± .19(−.47)		.041	0.49
Pycn. (24)	.957	.011	+ .023	11.48 ± .13(+.28)		.028	0.34
Single-strength grapefruit juice							
Refrac. (16)	.992	.009	+ .020	11.90 ± .11(+.24)		.023	0.28
Hydr. (16)	.969	.014	− .032	11.60 ± .18(−.38)		.034	0.41
Pycn. (16)	.961	.011	− .024	11.53 ± .13(−.29)		.029	0.35
Concentrated orange juice							
Refrac. (46)	.979	.007	+ .016		41.12 ± .29(+.67)	.016	0.67
Pycn. (12)	.969	.010	+ .021		40.70 ± .42(+.88)	.030	1.26
Concentrated grapefruit juice							
Refrac. (20)	.996	.007	− .015		41.33 ± .29(−.63)	.019	0.80
Pycn. (12)	.987	.013	− .022		41.45 ± .55(−.92)	.034	1.43

[a] All values enclosed in parentheses are maximum deviations.
[b] 95% confidence limit for prediction of soluble solids from a single Brix determination on similar samples by similar methods.

ble solids than were hydrometer and pycnometer values for all products, and were within 1% of soluble solids for all types of products except concentrated orange juice.

The average ratios (2nd column) are presented to the 3rd decimal place, which is significant in reporting the relationship of soluble solids to refractometer values of the samples studied. The ratios would be subject to some variation, however, if different samples were subjected to similar analyses. For instance, the soluble solids/refractometer value ratio of the fourteen 1959 orange concentrates was 0.984, and that of the earlier 23 4-fold orange juice concentrates was 0.975, whereas that of all the 46 orange concentrates was 0.979.

Table 4 has been set up to compare the accuracy and precision of three methods of estimating soluble solids in 4-fold concentrated orange juice from their refractometer values. The methods compared are 1) current industry practice of correcting refractometer values only for acid to degrees Brix, 2) correcting refractometer values for 7 components to soluble solids as described in the previous paper, and 3) converting refractometer values to soluble solids by use of the empirical factor 0.979 as derived above.

Since the 12 4-fold orange concentrates obtained in 1959 especially for this study were not analyzed for the various components affecting refractive index, only the 23 4-fold concentrates described in the previous paper were used for this comparison.

The 2nd column of Table 4 compares the accuracy of the 3 methods. For these 23 samples, soluble solids averaged 1.64° Brix lower than refractometer values corrected only for acid. Soluble solids averaged only about 0.2° Brix lower than refractometer values corrected for all components known to affect refractive index appreciably or converted by use of an empirical factor. The failure of the empirical conversion factor to produce an average identical with soluble solids is due to derivation of the conversion factor from 46 samples rather than the 23 involved in these calculations.

Table 4. Comparison of three methods for converting refractometer values of 4-fold concentrated orange juices to soluble solids.

Method	Sol solids in concentrate[a] (average) %	Observed range of sol. solids in concentrate[b] %	Std. dev. of observed sol. solids %
Refrac. value corr. for acid to ° Brix	40.36	39.94–40.82	0.257
Refrac. value corr. for 7 components to % sol. solids	41.79	41.45–42.13	0.250
Refrac. value × 0.979	41.83	41.37–42.29	0.255

[a] Average values of soluble solids by vacuum drying of concentrates reading 42.00° Brix by the methods indicated in column 1. These are obtained by multiplying 42.00 by the average ratio of observed soluble solids to observed degree Brix of 4-fold concentrates.

[b] Values obtained by multiplying 42.00 by minimum and maximum observed ratios.

The final two columns illustrate the relative precision of the three methods. There is little to choose among them. All are apparently limited by experimental errors of about the same magnitude.

Consideration was given to correcting refractometer values for acid, and developing a factor for converting the corrected values to soluble solids. The concentrated orange juices to which the procedure was applied had been blended to between 12.5 : 1 and 16 : 1 solids-acid ratios, and thus showed little variation in acid content. The factor developed for converting corrected refractometer values to soluble solids was 0.965, with a standard deviation of 0.008 and a maximum deviation of 0.031, which is no improvement in accuracy or precision over use of the conversion factor 0.979 applied directly to the refractometer values as read.

CONCLUSIONS

The average ratios presented in the 2nd column of Table 3 are suggested as factors for converting Brix values to soluble solids. Use of the factors for converting refractometer values to soluble solids resulted in considerably greater accuracy than the current practice of correcting refractometer values for acid to degrees Brix, and proved as accurate as correcting for 7 refractometrically active components, without loss of precision.

Should it be decided that an empirical conversion factor be adopted for refractometer values in estimating soluble solids, additional samples varying more widely in type and source should be examined. This may result in slight changes in the factor. The work done is sufficient to show that a factor could give a better estimation of soluble solids than do current procedures.

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Determination of Water-Holding Capacity of Ground Cooked Lean Meat [a]

(Manuscript received March 29, 1961)

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SUMMARY

A simplified hydraulic pressure method has been developed for determining the free moisture content of cooked meat. The effects of sample weight of the cooked meat, length of time of pressing, amount of pressure applied, and length of time of heating of the raw meat sample were evaluated. The technique was applied to determine the effects of ficin and salts (NaCl and $Na_5P_3O_{10}$) on the free moisture content of ground lean steak. A simple technique of determining drip loss on cooking is also described.

ONE OF THE most important features of meat is its ability to bind water. The amount of water bound is generally assumed to be determined by the degree of hydration of the proteinaceous material (Wierbicki *et al.*, 1956; Wierbicki and Deatherage, 1958; Hamm, 1959). Tenderness, juiciness, color, taste, shrinkage on cooking, and drip on freezing and thawing all appear to be directly related to the water-holding capacity of meat (Deatherage, 1955).

Since no methods are available for determining the hydration of the proteins of meat directly, the bound water content is obtained by subtracting free water content from total moisture content. The total moisture content is determined by the standard oven drying technique (e.g., 8 hr at 110°C). The hydraulic-pressure technique (Wierbicki and Deatherage, 1958; Grau and Hamm, 1953) is commonly used for determining the free moisture content of raw meat, and the centrifugal-force technique (Wierbicki *et al.*, 1957) is used for determining the free moisture content of heated and cooked meat. With these methods the amount of bound water found depends on the experimental conditions; they give only relative values. Nevertheless, for most purposes, it is sufficient to know relative differences in water-holding capacity as determined by a single method.

The hydraulic-pressure technique for raw meat is rapid and requires only 0.5 g for each determination. The centrifugal-force technique for heated and cooked meat, in contrast, requires a large sample for each determination (25 g), special glass centrifuge tubes, and an accurate control of the centrifugal force.

The present paper presents a hydraulic-pressure technique for determination of the free moisture content of ground cooked lean meat. The method requires only 1.5-g samples and only a few minutes for each assay. Evaluation is made of the effects of sample weight, press time, amount of pressure, and time of heating the raw meat sample. Also presented are applications of this method to lean ground steak meat with and without added ficin, sodium chloride, and sodium polyphosphate. A simple method is given for determining drip loss during cooking.

EXPERIMENTAL

The meat was trimmed of excess fat and passed through a food chopper four times using a pulverizer plate. Cooked meat was prepared by heating accurately weighed 10-g samples of freshly ground, lean steak 15 min in glass tubes (¾ × 5 in.) in boiling water. At the end of this period the temperature in the center of the meat reached 70°C. During the heat treatment the meat sample rested on a vertical 1-in. length of tygon tubing (¼-in. OD) so that the amount of drippings resulting from the heat treatment could be assessed. After the heat treatment, the meat sample was mashed with a fork and kept in another clean glass tube at 30°C until required for determination of free moisture content. A Carver press was equipped with a pressure gage from 0 to 600 psi, with increments of 10 psi. Quantities of the cooked meat weighed on an analytical balance and at room temperature (approx 24°C) were placed in a special pressure cell (Fig. 1) and compressed at selected pressures and periods. After the pressure was released the compressed sample was weighed and the free moisture content was calculated as the difference between the

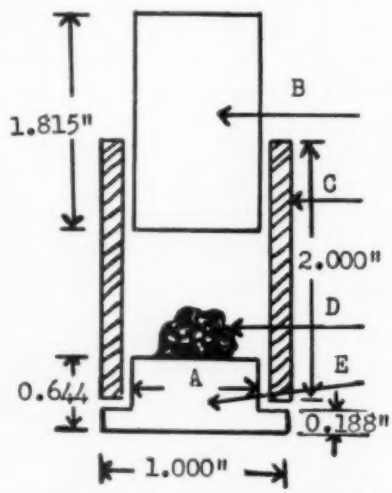

Fig. 1. Cross section of press cell; B) stainless-steel piston, diameter 0.778 in.; C) brass tube, 0.110 in. wall thickness, 0.780 in. ID; D) meat sample; and E) stainless-steel platform, diameter at A = 0.778 in.

[a] Contribution No. 129, Plant Research Institute, Research Branch, Canada Department of Agriculture, Ottawa.

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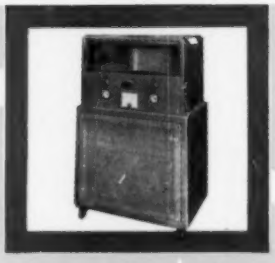

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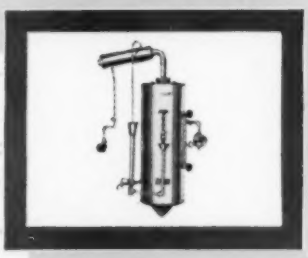

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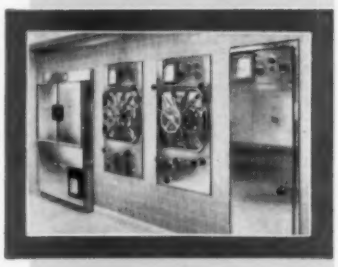

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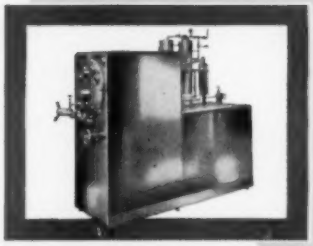

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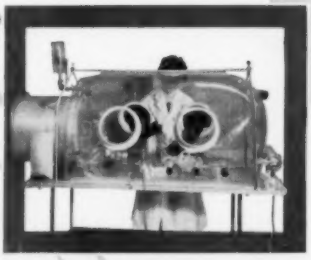

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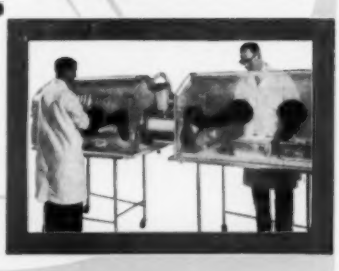

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weight of this sample before and after pressing. The free moisture content, as calculated, does not include water lost by drip during cooking.

Total moisture content of raw and cooked meat was determined by oven drying for 8 hr at 110°C.

RESULTS AND DISCUSSION

Table 1 summarizes the results of pressing cooked meat samples of various weights in the press cell for 60 sec at 500 psi. The data indicate good reproducibility within a sample range. When the sample increases from approx 0.5 to 1.5 g, the free moisture value decreases. When the sample range is 1.5–3.0 g, however, the free moisture values are consistent.

Table 1. Effect of weight of sample on the free moisture content of cooked meat.[a, b]

Meat sample	Av. sample weight[c] (before pressing) (g)	Av. free moisture (%)
A	0.4802	35.1 ± 1.0
	0.8784	31.8 ± 0.1
	1.8912	27.7 ± 0.8
	2.6356	28.0 ± 0.9
B	0.5258	35.7 ± 0.8
	1.0016	32.4 ± 0.5
	1.5318	30.8 ± 0.8
	2.0210	30.2 ± 0.8

[a] Drip loss during cooking (av. of four 10-g samples), respectively 21.7% and 21.5% for samples A and B, expressed on raw meat basis.
[b] Compressed in the hydraulic pressure cell for 60 sec at 500 psi.
[c] Results for sample A are averages of six samples for each weight group; those for sample B, of four samples for each weight group.

Table 2 shows the effect of length of time of pressing at 500 psi on approximately 1.5-g samples. The results indicate that the free moisture content is independent of press time after 1 min.

Table 2. Effect of pressing time on free moisture content of approx 1.5-g samples of cooked meat, compressed in the press cell at 500 psi.

Duration of pressing (sec)	Weight of sample before pressing (g)	Free moisture (%)
30	1.5206	26.2
	1.5354	26.8
60	1.4942	33.7
	1.5267	33.7
120	1.5000	34.4
	1.4983	33.6
180	1.4822	34.3
	1.4931	35.7
240	1.5222	33.8
	1.5466	34.3

Table 3 summarizes the free moisture values of approximately 1.5-g samples after 2 min at 300, 400, 500, or 600 psi. The results indicate no significant difference in varying the pressure from 300 to 600 psi.

Table 4 summarizes the drip losses of raw meat samples immediately after heating and the free moisture contents of the cooked samples as related to cooking time. The results indicate very little variation in either drip losses or free moisture contents from varied cooking time. However, there is a definite trend of increasing the drip losses of raw meat and decreasing the free moisture of the cooked meat with increasing the cooking time, giving a total moisture loss (drip and free moisture) of the same magnitude (52.2, 52.3, 52.3, and 52.6% respectively).

Table 3. Free moisture values of approx 1.5-g samples of cooked meat after 2 min of pressing at 300, 400, 500, or 600 psi.

Pressure (psi)	Weight of sample before pressing (g)	Free moisture (%)
300	1.4790	34.5
	1.5051	33.5
400	1.4859	33.2
	1.4974	34.9
500	1.4993	34.0
	1.4690	34.0
600	1.4988	34.2
	1.4850	34.3

Table 4. Drip losses immediately after heating, and the free moisture contents of the cooked samples as related to cooking time.

Cooking time of raw meat[a] (min)	Drip loss during cooking (%)	Free moisture[b] (%)
10	22.1	30.1
15	21.6	30.7
20	22.5	29.8
25	23.2	29.4

[a] Average of three 10-g samples.
[b] Average of two approx 1.5-g samples, compressed 2 min in the press cell at 500 psi.

The total solids content of the pressed-out juice of the cooked meat samples was between 2.5 and 3.5%. No correlation could be detected between pressed-out juice and any of the factors—size of sample, length of time of pressing, amount of pressure applied, and length of time of heating the raw meat.

The effects of proteolytic enzymes (papain and ficin) and salts (NaCl and $Na_5P_3O_{10}$) on the water-binding capacity of proteins have been studied by Whitaker (1961), Hamm (1959), and Wierbicki *et al.* (1957). The free moisture content of cooked meat after the addition of ficin and/or NaCl and $Na_5P_3O_{10}$ was determined with the press cell technique. Table 5 summarizes the drip losses of the treated samples immediately after cooking and the free moisture contents of the cooked samples as affected by length of incubation at 35°C with ficin or NaCl plus $Na_5P_3O_{10}$ or a combination of ficin and the salts. The results with the ficin-treated meat indicated a small reduction in drip losses, but corresponding slightly higher free moisture values. It must be noted that ficin caused breakdown of the raw meat proteins, and after 52 minutes of incubation at 35°C, small pieces of meat fiber became apparent in the free moisture squeezed out in the press cell. The salt treatments caused a large reduction in drip losses during cooking; the drip losses were not affected by length of incubation. Cursory organoleptic evaluation showed that the salt-treated samples were "rubbery" in texture after

Table 5. Drip losses of samples immediately after cooking, and the free moisture contents of the cooked samples as affected by the action of ficin, NaCl, and $Na_5P_3O_{10}$.

Meat sample	Treatment	Time at 35°C prior to cooking (min)	Drip loss during cooking[a] (%)	Free moisture[b] (%)
A	Control	....	36.4 ± 0.2	19.6 ± 0.1
	Ficin, 1.6×10^{-5} g per g of raw meat	19	31.6 ± 1.1	21.6 ± 0.6
		35	32.2 ± 1.3	20.3 ± 0.5
		52	28.9 ± 1.9	22.2 ± 0.3
B	Control	....	34.0 ± 0.3	....[c]
	Ficin, 1.6×10^{-5} g per g of raw meat	54	29.2 ± 0.8	....
		84	29.5 ± 0.6	....
		119	30.9 ± 0.6	....
		136	30.2 ± 0.9	....
		159	30.0 ± 0.5	....
	2% NaCl plus 2% $Na_5P_3O_{10}$	80	9.7 ± 0.5	....
		108	11.4 ± 0.1	....
		143	10.9 ± 0.7	....
	Ficin, 1.6×10^{-5} g per g of raw meat plus 2% NaCl plus 2% $Na_5P_3O_{10}$	71	10.2 ± 0.7	....
		106	9.3 ± 1.3	....
		123	9.4 ± 1.1	....
		146	9.6 ± 1.3	....
C	Control	....	30.6 ± 0.2	20.5 ± 0.2
	2% NaCl plus 2% $Na_5P_3O_{10}$	20	9.8 ± 0.1	16.1 ± 0.3
		50	9.4 ± 0.4	15.7 ± 0.1
		80	9.9 ± 0.6	13.8 ± 0.9

[a] Expressed on raw meat weight basis.
[b] Approx 1.5-g samples pressed 2 min at 500 psi, expressed on cooked meat weight basis.
[c] Not done.

cooking, but the samples containing both ficin and salts had a normal texture.

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Rheological Methods for Studying the Physical Properties of Emulsifier Films at the Oil-Water Interface in Ice Cream

(Manuscript received April 3, 1961)

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SUMMARY

Information concerning the physical nature of emulsifier films adsorbed at the oil-water interface in emulsions can be derived by studying emulsion flow properties, with particular reference to their dependence on mean globule size and internal phase viscosity. The structure of emulsifier complex at the oil-water interface in ice cream emulsions has been examined in this way.

The viscosity data indicate that the fat globules are surrounded by a rigid film, so that the flow properties of the emulsions resemble those of suspensions of solid spheres in liquid media. The milk proteins are involved in the development of the rigid layer at the oil-water interface, probably through lipoprotein complex formation. The rigid interfacial layer, which may be hydrated, is of substantial thickness with respect to the dimensions of the oil globules.

SEVERAL METHODS are available for study of the properties of emulsifier films at the oil-water interface. They fall into the following categories:

(a) Determination of force-area relationships by the Langmuir-Adams trough technique. (Rideal, 1936; Adam, 1941; Harkins, 1952).

(b) Calculation of force-area relationships from the change of interfacial tension with concentration of emulsifier (Alexander and Johnson, 1950).

(c) Determination of surface viscosity, elasticity, and rigidity from the damping effect exerted by the interfacial film of emulsifier on an oscillating pendulum system (Blakey and Lawrence, 1954; Boyd and Vaslow, 1958; Bulas and Kumins, 1958; Criddle and

Meader, 1955; Neilson *et al.*, 1958; Tachibana and Inokuchi, 1953). Certain corrections need to be applied to the experimental data (Kestin and Wang, 1957; Tschoegl, 1958).

(d) Measurement of "two-dimensional viscosity" by use of the concentric cylinder viscometer principle (Bernard, 1957; Davies and Mayers, 1960; Ellis *et al.*, 1955), or a canal viscometer (Joly, 1939, 1947).

(e) Qualitative assessment of the strength of interfacial films (Serrallach *et al.*, 1933), or the force required to rupture the film when subjected to torsional shear (Buzagh and Szonyi, 1949).

These techniques can be applied only to model systems in which extended, flat, stationary oil-water interfaces are subjected generally to slight deformations.

In practice it is not easy to explain the rheological behavior of emulsions in terms of observations on such systems. Indeed, little attempt has been made to do so. A very appropriate description of the present situation (Sumner, 1960) reads "The approach to the properties of emulsions as dispersions has been largely physical and mathematical, and interactions between droplets have normally been ignored. It is clear that progress in emulsion theory will require investigation not only of the properties of interfacial films as such, but of their influence on the mutual relations of the film covered droplets."

Interpretation of data derived by the above methods requires consideration of aging effects. In many instances, depending on the concentration of emulsifier employed, relatively long periods are required for interfacial films to develop their maximum viscosity, plasticity, etc. (Serrallach *et al.*, 1933; Sherman, 1953).

Some of the techniques can be applied only if the chemical constitution and purity of the materials involved are known with certainty. Satisfactory interpretation of data depends on this also. For many emulsion products of commercial interest, e.g., ice cream, this is not possible, since the chemical constitution of the emulsifier, and other raw ingredients—milk powder, stabilizer, etc.—are not known with any degree of accuracy.

It appears desirable to devise alternative methods for studying the properties of emulsifier films at the oil-water interface in emulsions.

Two methods are proposed. They allow for interaction between globules, the interaction between interfacial films around adjacent globules, the influence of the interfacial film on fluid circulation within the globules and on the deformability of the globules, and influence of film hydration and electro-viscous effects on emulsion viscosity. The proposed methods involve study of the influence of globule mean volume diameter (d_v) and internal phase viscosity (η_i) on emulsion viscosity at such rates of shear that emulsion viscosity (η) is independent of shear rate.

THEORY OF PROPOSED METHODS

Influence of d_v on η. For emulsions with narrow globule size distributions in which d_v does not exceed 5 μ, the influence of d_v on η depends on the state of the emulsifier film at the oil-water interface (Sherman, 1960).

If the interfacial film exhibits the properties of a plastic solid, decreasing d_v—by further homogenization, or homogenization at higher pressure—leads to an increase in η. When d_v falls below 2 μ the effect is very pronounced. The $\eta - d_v$ relationship is curvilinear and is observed over a wide range of dispersed phase volume concentrations (ϕ).

When the interfacial film is fluid, and does not prevent the transmission of shear effects to the globules, decreasing d_v exerts no effect on η until ϕ approaches 0.5. Then η increases linearly with decreasing d_v. The higher the value of ϕ the more pronounced is the effect on η.

At the rates of shear at which η is measured the globules are completely deflocculated, i.e. there is no internal-structure build-up due to aggregation and formation of links between globules, and η depends on hydrodynamic interaction between globules only.

For emulsions that achieve optimum, dodecahedral, packing at a ϕ value of 0.74 approximately, and in which the globules behave as rigid particles,

$$a_m = d_v \left(3\sqrt{\frac{0.74}{\phi}} - 1 \right) \qquad [1]$$

where a_m is the minimum mean distance between adjacent globules (Sherman, 1960). Thus, d_v exerts a more pronounced effect on a_m than does ϕ. Emulsion viscosity is related exponentially to a_m.

In emulsions where the interfacial film is fluid, the globules undergo distortion at all but low rates of shear. This will affect a_m. The degree of deformation will depend partially on ϕ, so that in closely packed systems little deformation is possible. However, at high values of ϕ, e.g., 0.7, small deformations reduce η to a much greater extent than large deformations at ϕ values around 0.5–0.6.

Influence of η_i on η. If the interfacial film is fluid it is to be expected that η_i will influence η. When the interfacial film is solid the globules will behave as rigid spheres, i.e., as if $\eta_i \to \infty$. In the latter case η_i will not affect η.

Several modifications of Einstein's (1911) viscosity equation for very dilute suspensions of rigid spheres have been proposed (Taylor, 1932; Leviton and Leighton, 1936; Oldroyd, 1955; Rajagopal, 1960) to account for the influence of η_i on η in certain emulsions.

When studying the effect of η of variation in η_i care must be taken to ensure that the interfacial equilibrium remains unaltered (Sherman, 1955).

EXPERIMENTAL METHODS

Discussion in the theoretical section indicates that the structure of the emulsifier film around globules in an emulsion can be deduced indirectly from viscosity data, provided one knows the influence of d_v and η_i on η.

These methods have been applied to a study of interfacial structure in ice cream mixes (dilute oil-in-water emulsions).

Influence of d_v on η. Ice cream mixes were prepared in Pilot Scale Plant according to the recipes:

	% composition (w/w)			
	(a)	(b)	(c)	(d)
Oil phase				
Hardened vegetable fat (iodine value 70, mp 33–34°)	4.35	8.38	12.41	15.54
Commercial-grade glyceryl monostearate (approx. 30% monoglyceride)	0.53	0.53	0.53	0.53
Aqueous phase				
Water	66.24	62.21	58.15	55.05
Milk powder	13.10	13.10	13.10	13.10
Sugar	15.60	15.60	15.60	15.60
Vegetable gum	0.18	0.18	0.18	0.18
ϕ	0.053	0.101	0.148	0.184

Five mixes were prepared in accordance with each recipe, and respectively homogenized at 1000, 1500, 2000, 2500, and 3000 psi.

Globule size distribution, and d_v of each emulsion, were determined from microscopic counts on approximately 2,000 globules per sample.

$$d_v = 3\sqrt{\frac{n_1 d_1^3 + n_2 d_2^3 \text{ ——— } + n_x d_x^3}{n_1 + n_2 \text{ ——— } n_x}} = 3\sqrt{\frac{\Sigma n d^3}{\Sigma n}} \qquad [2]$$

where n_1, n_2, $---$ n_x, are the respective numbers of globules with diameters d_1, d_2, $---$ d_x.

Of the globules, 95% or more fell within the size range 0.5 2.5 μ. Most of these were within the limits 0.5–1.5 μ. Homogenization of the mixes for any one recipe at progressively higher pressures shifted the size distribution from the upper to the lower limit.

Influence of η_i on η. Two series of emulsions were prepared.

(1) Ice cream type of recipe

Oil phase	% composition (w/w)
Hardened vegetable fat	7.43–48.06
Molecularly distilled glyceryl monostearate (soap-free, or with potassium stearate)	0.18
Aqueous phase	
Water	63.57–22.94
Spray-dried milk powder	13.10
Sugar	15.65

The two phases were heated independently to 70°C and then mixed with vigorous agitation, the oil phase being dispersed in the aqueous phase. Finally the crude emulsion was passed several times through a laboratory-model homogenizer.

(2) Liquid paraffin-in-water emulsions:

Medicinal-grade liquid paraffin was emulsified in water using 0.2% molecularly distilled glyceryl monostearate (soap-free, or with potassium stearate; expressed as % of emulsion total weight). The volume concentration of oil phase ranged from 0.04 to 0.35.

The glyceryl monostearate was dissolved in the oil phase. Both phases were heated independently to 70°C, and the oil phase then added with vigorous agitation to the aqueous phase. The crude emulsion was then passed several times through a laboratory homogenizer.

All viscosities (η, η_0, and η_i) were determined with a concentric-cylinder Couette-type viscometer (McKennell, 1960). This was calibrated previously against silicone oils covering a wide range of viscosities. The rates of shear over which viscosities were examined extended from 60–800 $sees^{-1}$.

Most of the emulsions exhibited non-Newtonian flow.

DISCUSSION OF RESULTS

Influence of d_v on η. Fig. 1 shows linear $\eta - \frac{1}{d_v}$ relationship for the viscosity data of the four ice cream recipes (Table 1).

For the two lowest ϕ values, decreasing d_v produces little change in η over the d_v range examined. This does not apply to the other two mixes. In both cases η increases curvilinearly with decreasing d_v. Increase in η per unit change of d_v is greater at a ϕ value of 0.184 than at 0.148. The reason becomes apparent in later discussion.

The viscosities of the ice cream mixes follow the relationship:

$$\eta = m\left(\frac{1}{d_v}\right) + C \qquad [3]$$

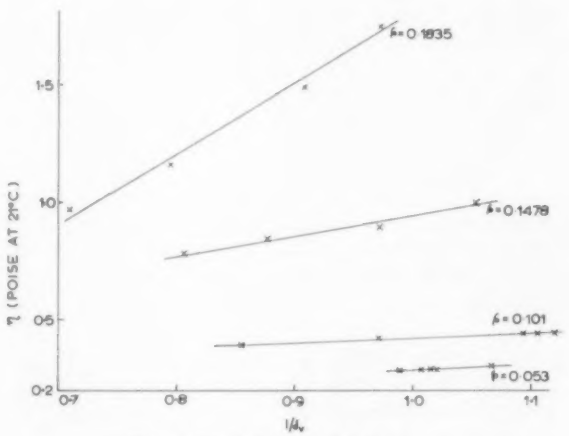

Fig. 1. The influence of $1/d_v$ on η.

Table 1. Rheological data for ice cream mixes prepared on pilot plant.*

ϕ	Emulsion viscosity (poises @ 21°C) Experimental (η_e)	Calculated (η) From [a]	Calculated (η) From [g]	Continuous-phase viscosity (η_0) poises @ 21°C)	Mean globule diameter d_v (μ)	Interglobular separation a_m (μ) Calculated from [1]	Interglobular separation a_m (μ) Amended from [i] and [e] to allow for Δr	Thickness of hydrated layer Δr (μ) Calculated from [f]	Thickness of hydrated layer Δr (μ) Calculated from [i] and [e]
	0.285				1.014	1.434	0.834	0.621	0.305
	0.286			0.109	0.992	1.403	0.803		
0.0526	0.290	0.124	0.128		0.985	1.392	0.792		
	0.293				0.980	1.385	0.785		
	0.31				0.937	1.325	0.725	0.659	0.300
	0.39				1.170	1.105	0.505	0.649	0.270
	0.42	0.146	0.167	0.117	1.027	0.971	0.371		
0.1010	0.44				0.914	0.864	0.264		
	0.44				0.904	0.854	0.254		
	0.44				0.892	0.843	0.243	0.549	0.230
	0.78				1.240	0.882	0.282		0.339
	0.84				1.114	0.792	0.192		
0.1478	0.89		0.203	0.121	1.028	0.731	0.131		
	0.99				0.949	0.675	0.075		0.304
	1.29				0.946	0.673	0.073		
	0.97				1.413	0.835	0.235		0.346
	1.10				1.308	0.773	0.173		
0.1835	1.16		0.250	0.129	1.259	0.744	0.144		
	1.48				1.100	0.650	0.050		
	1.74				1.028	0.607	0.007		0.342

* The *numbers* in square brackets refer to equations quoted in the paper. The *letters* in square brackets refer to equations quoted in Appendix I.

wine, honey, mushroom, sea-food,
smoked ham, chicken bouillon, guava

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a progress report on "Immersion Freezing" from Airco

The use of liquefied gases as freezing agents opens impressive new possibilities for progress in the food industry. "Immersion Freezing", as this process is known, holds promise of faster production, higher quality standards and the availability of totally new food products.

"Immersion Freezing" has received much publicity which, like many new major technological breakthroughs, is accompanied by rumors and claims that tend to distort the picture. The purpose of this report is to provide a factual evaluation of the process and its potential.

Air Reduction Sales Company, through its Cryogenic and Special Products Departments and its Customer Service Laboratory, has been a pioneer in "Immersion Freezing". The facts in this report are based on two years of development and evaluation. We trust this report will provide the reader with reliable information that will enable him to evaluate the potential of "Immersion Freezing" in relation to his own operation.

"Immersion Freezing" is an almost instantaneous freezing process which involves the immersion of food products into a liquefied gas. It requires custom designed equipment. "Immersion Freezing" with liquefied gases is going to be more expensive in unit freezing costs than conventional freezing techniques. This higher basic cost, however, may be more than offset by what Airco technologists call the XYZ factors.

X—Quality improvement
Y—Increased production
Z—New product possibilities

FACTOR X

Represents improvement in quality. Quality to the frozen merchandiser might be expressed in improvements in flavor, color, vitamin retention, tenderness . . . resulting in better over-all consumer acceptability.

For example:

1. Strawberries frozen with liquefied gases retain normal color and close-to-normal texture.
2. Meat has better color and is more tender.
3. Whole kernel corn now has no loss in kernel constituents or vitamins, on-the-cob flavor is retained.
4. Ice cream has more uniform texture and appearance.

FACTOR Y

Represents improvement in production or throughput. The use of liquefied gases as expendable refrigerants allows the food freezer to supplement existing refrigeration for peak seasonal loads. Improvements in throughput may provide many savings in processing to the frozen food packer.

For example:

1. The hardening room can be by-passed in the freezing of ice cream.
2. A shorter freezing cycle allows reduction in the floor space requirements in many processes.
3. The freezing of cut-up poultry can be a continuous process, by-passing freezing storage and extra handling.
4. In the freezing of whole kernel corn, yield may be increased by as much as 25%.

FACTOR Z

Represents the ability to freeze products which cannot be successfully frozen by any other means. Following are some of the advances made by Airco in this all-important area of new product development:

1. CITRUS CELLS—After immersion of a peeled orange, grapefruit, lemon, lime or other citrus fruit in a liquefied gas, the product can be reduced to individual citrus cells. These individual citrus cells can be used in a variety of applications—ice cream, cottage cheese, confectionery products, orange juice concentrate.
2. TOMATO SLICES—"ImmersionFreezing" of tomato slices or wedges yields a highly acceptable product. The extremely fast freezing results in a product which, when thawed, closely approximates the fresh tomato.
3. SLICED BANANAS—Bananas frozen by immersion in liquefied gases retain a high percentage of their original appearance, color and texture.

All aspects of "Immersion Freezing" covered by Airco's XYZ factors are in various stages of development by Air Reduction's Customer Service Laboratory. Only one of them is in commercial production at this time. They are listed here to demonstrate the possibilities for better quality, production savings and new products which may offset the higher initial cost.

DEVELOPMENT OF DATA:

The development of sound technical and engineering data is the basis of the Air Reduction approach to this field. Following is information based on Airco studies covering areas where care must be exercised in the application of "Immersion Freezing".

1. Liquid nitrogen is not an effective bactericidal agent.
2. Pre-cooling or other forms of treatment are sometimes required prior to "Immersion Freezing".
3. Some packaging materials, adhesives, glues, etc., will not withstand the extremely low temperatures resulting from liquefied gas freezing.
4. Enzyme systems appear to function even at these low temperatures.
5. Each product requires individual evaluation for freezing by this method.

SPECIAL EQUIPMENT NEEDED:

The Special Products Department of Air Reduction Sales Company is developing the freezing equipment for "Immersion Freezing". Each product will require custom designed equipment for freezing. Design considerations include throughput requirements, product characteristics, handling equipment and packaging requirements.

The Cryogenic Engineering Department of Air Reduction is developing closed cycle refrigeration systems using either liquid nitrogen or nitrous oxide. These systems will take into account the normal operation of the producer's plant, seasonal characteristics of growing or harvesting of product, stability of the market, etc. Capital requirements for this type of freezing system are considerable and, therefore, long-term use agreements with adequate guarantees and financing will be required.

Readers are invited to write to Air Reduction Sales Company for additional information, or for advice concerning specific freezing problems.

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where m is the gradient, and C the intercept on the ordinate axis. The gradient m increases curvilinearly with increasing ϕ; C increases linearly with increasing ϕ for the ϕ values examined. In general, C is related exponentially to ϕ (Sherman, 1960).

From [1], $\eta - a_m$ relationships have been calculated (Fig. 2). This form of presentation has the advantage that all data are summarized on a single graph. It also suggests an explanation for the experimental observations since η increases curvilinearly as a_m decreases. When a_m falls below approximately 1 μ, η increases more pronouncedly with further decrease in a_m. This critical value of a_m is rather high. It suggests that some additional phenomenon is involved that has not been considered hitherto.

The most likely explanation would be hydration of milk protein adsorbed at the oil-water interface, and/or the development of an extensive layer of surface-denatured and coagulated protein around the globules.

Appendix 1 indicates how the thickness of this layer (Δr) can be calculated theoretically from viscosity and mean globule-size data, starting with the Einstein (1911) equation for dilute suspensions of solid spherical particles [a]. The Guth-Simha (1936) extension of this equation to more concentrated systems [g] is also considered. This latter equation allows for interaction between the particles at higher values of ϕ by introducing a factor 14.1 ϕ^2. Several workers believe that a lower value of ϕ^2 should be employed (Sherman, 1959).

Calculated values of Δr are quoted in Table 1, which also contains all other relevant data. On the basis of the Guth-Simha equation the $\eta - a_m$ relationships have been amended to give the broken-line curve shown in Fig. 2. An average thickness of 0.3 μ is assumed for Δr. In the amended form the curve indicates that η increases more pronouncedly when a_m falls below 0.5 μ. This agrees with previous observations (Sherman, 1960).

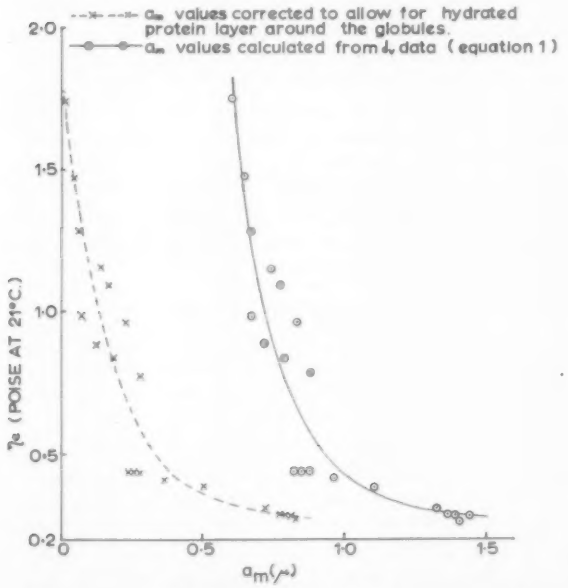

Fig. 2. The influence of interglobular separation (a_m) on the viscosity of ice cream mix (η_0).

Ice cream mixes, although relatively dilute emulsions, show marked rise in η with change in d_v because the globules are surrounded by a "shell" 0.3 μ thick. This "shell" alters the effective dimensions of the globules. It increases the interaction between the globules at relatively large values of a_m.

Interpretation of viscosity data in this way so as to show the dependence of η on a_m indicates also why Einstein's equation requires no term for particle size. For the values of ϕ over which this equation holds, the values of a_m are so large that η remains unaffected by change in d_v. Since it is assumed that there is no interaction between the spherical particles and the continuous medium, no "shell" of thickness Δr develops around the particles. Disagreement regarding the factor 14.1 ϕ^2 in the modified form of this equation may be due to disregard of the influence exerted by d_v at higher values of ϕ, particularly if the sphere diameters are on the order of 1 μ or less.

Fig. 1 indicates some further points of interest arising from the dependence of η on d_v. At higher values of ϕ, larger homogenization pressures are required to achieve the same d_v. As an example of this we derive the homogenization pressures that have to be applied to obtain a d_v of 1.0 μ in ice cream mixes with different fat contents by replotting the Fig. 1 data as d_v against homogenization pressure.

% fat content	Homogenization pressures (psi) required to achieve d_v of 1.0 μ
4.35	1,500
8.38	1,600
12.41	2,200
15.54	2,800

Since mixes of lower fat content exhibit lower d_v values than those of high fat content, increasing the homogenization pressure from 1000 psi to 3,000 psi reduces d_v more in the latter than in the former. Ice cream mixes containing only 4% fat are influenced little by increased homogenization pressure.

% fat content	d_v (μ) at following homogenization pressures (psi):	
	1,000	3,000
4.35	1.01	0.94
8.38	1.17	0.84
12.41	1.24	0.95
15.54	1.41	1.03

Influence of η_1 on η. Tables 2 and 3 respectively show viscosity data for liquid paraffin-in-water and ice cream emulsions containing molecularly distilled glyceryl monostearate as emulsifier. The viscosities determined experimentally are quoted; also viscosities calculated by means of the Taylor (1932) and Leviton and Leighton (1936) equations.

For the liquid paraffin-in-water emulsions the calculated viscosity values agree well with those found experimentally for values of ϕ extending to approx. 0.2. Up to a ϕ value of 0.15, Taylor's (1932) equation

holds; at a ϕ value of 0.2, Leviton and Leighton's modification shows better agreement with the experimental values. At higher ϕ values the deviations between theoretical and experimental values of η become quite pronounced.

Table 2. Viscosity data for liquid paraffin-in-water emulsions emulsified with molecularly distilled glyceryl monostearate.

Emulsifying agent	ϕ	η_i (poises at 70°C)	η_o (poises at 70°C)	η (poises at 70°C) Measured	Calculated from [3][a]	Calculated from [4][a]
Monoglyceride plus 6.0% potassium stearate	0.0424	0.164	0.0045	0.0049	0.0050	0.0051
	0.0825	0.164	0.0045	0.0052	0.0054	0.0057
	0.1523	0.159	0.0045	0.0062	0.0062	0.0073
Soap-free monoglyceride	0.0424	0.167	0.0045	0.0052	0.0050	0.0051
	0.0825	0.160	0.0045	0.0056	0.0054	0.0057
	0.1523	0.160	0.0045	0.0066	0.0062	0.0073
	0.2000	0.161	0.0045	0.0086	0.0067	0.0088
	0.2560	0.161	0.0045	0.0092	0.0073	0.0111
	0.3470	0.161	0.0045	0.0220	0.0083	0.0176

[a] Refers to equations quoted in the text.

Thus the interfacial layer surrounding liquid paraffin-in-water emulsions stabilized by molecularly distilled glyceryl monostearate is in the fluid state.

Similar agreement between experimental and theoretical values of η is not found with ice cream emulsions, even for the values of ϕ extending to 0.2. In all instances but one, the experimental values of η are higher than those calculated. This indicates, in agreement with the comments of the previous section, that the globules are surrounded by a rigid layer and behave as solid spheres. The major contributor to this rigid layer must be the protein content of the milk powder.

Further support for this conclusion was obtained from unpublished interfacial tension data (Sherman). Interfacial tensions were determined at 70°C by the drop-volume method (Harkins and Humphery, 1916) for solutions of commercial and molecularly distilled grades of glyceryl monostearate in hardened vegetable fat against water, or water plus milk powder. The reduction in interfacial tension, i.e., increase in surface pressure, was always much greater when milk powder was present in the aqueous phase.

Table 3. Viscosity data for ice-cream-type mixes emulsified with molecularly distilled glyceryl monostearate.

Emulsifying agent	ϕ	η_i (poises at 70°C)	η_o (poises at 70°C)	η (poises at 70°C) Measured	Calculated from [3][a]	Calculated from [4][a]
Soap-free monoglyceride	0.0825	0.176	0.019	0.023	0.023	0.024
	0.1523	0.171	0.019	0.037	0.026	0.031
	0.2560	0.180	0.019	0.050	0.030	0.045
	0.347	0.180	0.019	0.090	0.035	0.070
	0.436	0.172	0.019	0.30	0.039	0.107
	0.534	0.173	0.019	1.80	0.043	0.193
Monoglyceride plus 3.0% potassium stearate	0.1523	0.178	0.019	0.035	0.025	0.031
Monoglyceride plus 6.0% potassium stearate	0.0825	0.244	0.019	0.025	0.022	0.024
	0.1523	0.181	0.019	0.035	0.026	0.031
	0.347	0.175	0.019	0.122	0.035	0.070
	0.438	0.173	0.019	0.26	0.040	0.119
	0.534	0.180	0.019	1.75	0.043	0.194
Monoglyceride plus 7.0% potassium stearate	0.1534	0.181	0.019	0.034	0.026	0.030

[a] Refers to equations quoted in the text.

These changes in interfacial tension can be attributed to complex formation. This follows penetration of the emulsifier film around the oil globules by the non-polar side groups of the first layer of adsorbed milk protein (Matalon and Schulman, 1949; Eley and Hodge, 1957). Since the concentration of milk powder in the water phase was kept constant, the magnitude of interaction depended on the quantity of unassociated molecules of emulsifier at the interface.

"Creaming" tests with ice cream emulsions at 20°C indicate that as much as 80–90% of the total protein content migrates to the cream layer. Thus, the lipoprotein complex at the oil-water interface may be associated with a layer of denatured protein. The thickness of an adsorbed monomolecular protein film should not exceed 0.003 μ so that this layer of denatured protein may be of polymolecular dimensions.

It is conceivable that the many films of protein within the protein layer have undergone different degrees of denaturation and coagulation, depending on their proximity to the oil-water interface. The magnitude of these changes would govern the amount of water bound by the protein films.

Bulk hardened vegetable fat with commercial or molecularly distilled grades of glyceryl monostearate dissolved therein, begins to crystallize within the temperature range 23.9–22.2°C, as found by dilatometry. Emulsified hardened vegetable fat supercools, and crystallization begins at approx. 15°C. Since all viscosities in the present work were determined at 21°C, it can be taken that under the test conditions the oil within the globules was liquid. The globules behave as rigid spheres because they are enveloped in a solid layer of lipoprotein.

Previous work (Sherman, 1960) suggests that for emulsions stabilized by non-ionic emulsifiers interfacial rigidity is found only in the water-in-oil systems. These observations were fortuitous, arising from the choice of emulsifiers. The present work shows that interfacial rigidity can be obtained in oil-in-water emulsions also as a result of complex formation at the interface. Thus, the proposition that only water-in-oil emulsions are stabilized by rigid interfacial films (Schulman and Cockbain, 1940a, b) does not find general application.

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The author thanks the boards of Unilever Ltd., and T. Wall & Sons (Ice Cream) Ltd. for permission to publish this paper.

APPENDIX I

Methods for Calculation of Hydration Layer Around the Globules in Ice Cream Emulsions

For very dilute suspensions of solid spherical particles dispersed in a fluid medium, the Einstein (1911) equation states that:

$$\eta = \eta_o [1 + 2.5 \phi], \text{ or } \frac{\eta - \eta_o}{\eta_o} = \eta_{sp} = 2.5 \phi \quad [a]$$

If the globules are surrounded by a hydration layer

$$\eta_e = \eta_o\,[1 + 2.5\,(X\phi)\,],\ \text{or}\ \frac{\eta_e - \eta_o}{\eta_o} = 2.5\,X\phi \quad [b]$$

where η_e is the experimentally determined emulsion viscosity, and X is the apparent increase in ϕ.

$$\text{Therefore,}\quad \frac{\eta_e - \eta_o}{\eta - \eta_o} = X \quad [c]$$

$$\text{Also,}\quad X = \frac{\Delta\phi + \phi}{\phi} = \frac{N^4/3\pi\,(r + \Delta r)^3}{N^4/3\pi r^3} = \frac{(r + \Delta r)^3}{r^3} \quad [d]$$

where N is the number of globules per unit volume of emulsion.

$$\text{So,}\quad \Delta r = \sqrt[3]{X}\,r - r = r(\sqrt[3]{X} - 1) \quad [e]$$

From, [c] and [e]

$$\Delta r = r\left[3\sqrt{\frac{\eta_e - \eta_o}{\eta - \eta_o}} - 1\right] \quad [f]$$

For higher values of ϕ than those covered by [a], the Guth-Simha modification (Guth and Simha, 1936) of [a] is applied:

$$\eta = \eta_o[1 + 2.5\phi + 14.1\phi^2],\ \text{or}\ \frac{\eta - \eta_o}{\eta_o} = 2.5\phi + 14.1\phi^2 \quad [g]$$

If the globules are surrounded by a hydrated layer

$$\eta_e = \eta_o\,[1 + 2.5\,(X\phi) + 14.1\,(X\phi)^2],$$

$$\text{or}\ \frac{\eta_e - \eta_o}{\eta_o} = 2.5\,X\phi + 14.1\,(X\phi)^2 \quad [h]$$

$$\text{Therefore}\quad \frac{\eta_e - \eta_o}{\eta_e - \eta_o} = \frac{2.5\,X + 14.1\,X^2\phi}{2.5 + 14.1\,\phi}$$

$$14.1\,\phi\,X^2 + 2.5\,X - \left[(2.5 + 14.1\,\phi)\left(\frac{\eta_e - \eta_o}{\eta - \eta_o}\right)\right] = 0 \quad [i]$$

This equation can be solved for X since all values are known with the exception of X.

In conjunction with [e], Δr can be determined.

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Variations in Heat Treatment of Individual Product Particles in Plate Heat Exchangers

(Manuscript received May 1, 1961)

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SUMMARY

Variation in heat treatment of individual particles during processing of certain foods is important in relation to destruction of enzymes and microorganisms, and changes in flavor, color, or physical properties of the product. Possible extremes of heat treatment during processing in three plate heat exchangers were evaluated. The results were markedly influenced by the sensitivity of methods for detecting the slowest and fastest particles. A conductivity test for residence times detected variations of heat treatment of about threefold in a small proportion of the test fluid in two of the exchangers, and a motion-picture procedure showed a similar range in one unit. Other evidence indicated that small amounts of fluid received much greater variations in heat treatment, especially under unfavorable conditions of equipment operation. Fastest and slowest particles are necessarily defined arbitrarily on the basis of the sensitivity of the measuring system, and hence may imply unwarranted confidence in the lethality of heat treatment of the fastest particles. Design and operation of plate heat exchangers should avoid low-velocity zones and air-pocket formation, since these may contribute to fouling of heat-transfer surfaces, build-up of thermophilic organisms, and flavor damage to the product.

IN HIGH-TEMPERATURE SHORT-TIME (HTST) sterilization and ultra-high-temperature (UHT) pasteurization processes, the heat treatment given individual product particles during passage through heating or cooling equipment may vary considerably. Nevertheless, uniform heat treatment is desired: The minimum treatment is important in destruction of pathogens, spoilage organisms, and certain enzymes; the maximum treatment may influence flavor, color, or other properties of the product, or the amount of material cooked on heat-transfer surfaces.

Heat treatment depends on both temperature and time of holding. One approach to achieving uniform residence time of particles in equipment is to promote effective continuous mixing of product during flow, which also assures uniform temperature treatment. Hence, uniformity of residence time among individual particles is an important criterion of the suitability of a heat exchanger or holding device for food products.

This paper deals with variations in heat treatment of individual product particles in three plate heat exchangers, particularly in relation to varied residence times.

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LITERATURE REVIEW

Walzholz (1940) studied the flow characteristics of holding devices by recording conductivity changes as a dilute salt solution replaced water (or *vice versa*) during flow through equipment. He obtained S-shaped curves representing the residence-time distribution of individual particles. The initial change in conductivity was abrupt, and provided a measure of shortest residence time—the most important point on the curve from a bacteriological viewpoint. The conductivity approached its final value asymptotically, so Walzholz arbitrarily adopted the point at which 95% of the product had passed through the apparatus as a measure of the longest residence time—the most important point in relation to physical properties of the product. The ratio of the longest to the shortest residence times, termed "Gutegrad," was used as an important criterion of uniformity of residence time in the holding device.

The term "efficiency" is (unfortunately) in common use in the United States and Great Britain to express uniformity of residence times. It is defined as the ratio of the time for the fastest observable particle to an average residence time (Cuttell, 1948; Jordan *et al.*, 1952). This criterion emphasizes the importance of the shortest residence time without consideration of extremes of overholding.

Data on flow patterns, pressure drops, and heat-transfer characteristics of the three plate heat exchangers used in the present study are reported elsewhere (Watson *et al.*, 1960; McKillop and Dunkley, 1960). Flow characteristics were studied in one unit by observation through a clear plastic window, and in all three exchangers by the Walzholz method.

One of the plate exchangers (unit A) had flat heat-transfer plates alternated with corrugated, perforated, gasketed plates used to secure desired clearances and direct the flow in the space between adjacent heat-transfer plates. In this unit it was convenient to replace an individual heat-transfer plate with a transparent Plexiglas plate for observation or photographing of flow. Motion pictures were used to record confluence of a safranin solution as it displaced water in the space between the plates. From these pictures it was possible to draw velocity profiles, calculate "shortest" and "longest" residence times of dye, and observe behavior of air pockets trapped in the flow channel. Variations in residence times of individual particles passing through a single plate of unit A were markedly influenced by three flow behaviors. A relatively unobstructed channel just inside the gaskets allowed higher velocities of small streams near the edges of the flow channel. Low-velocity zones permitted small spots of dye to persist in the flow area for a substantial time after the principal flow channels were flushed free of dye. In downflow passes at low flow rates, air was trapped in pockets in the flow channel, thereby reducing residence times.

METHODS

The equipment and methods have been described (Watson *et al.*, 1960). Some terms used in reporting and discussing the results are defined arbitrarily as follows:

Fastest particle. Measured as the first response detected by the method specified (conductivity or motion picture).

Slowest particle. 1) By the conductivity method calculated from the time required for 98% of the particles to pass the outlet electrode; 2) by motion picture method calculated from line of demarcation between dye and water when safranin solu-

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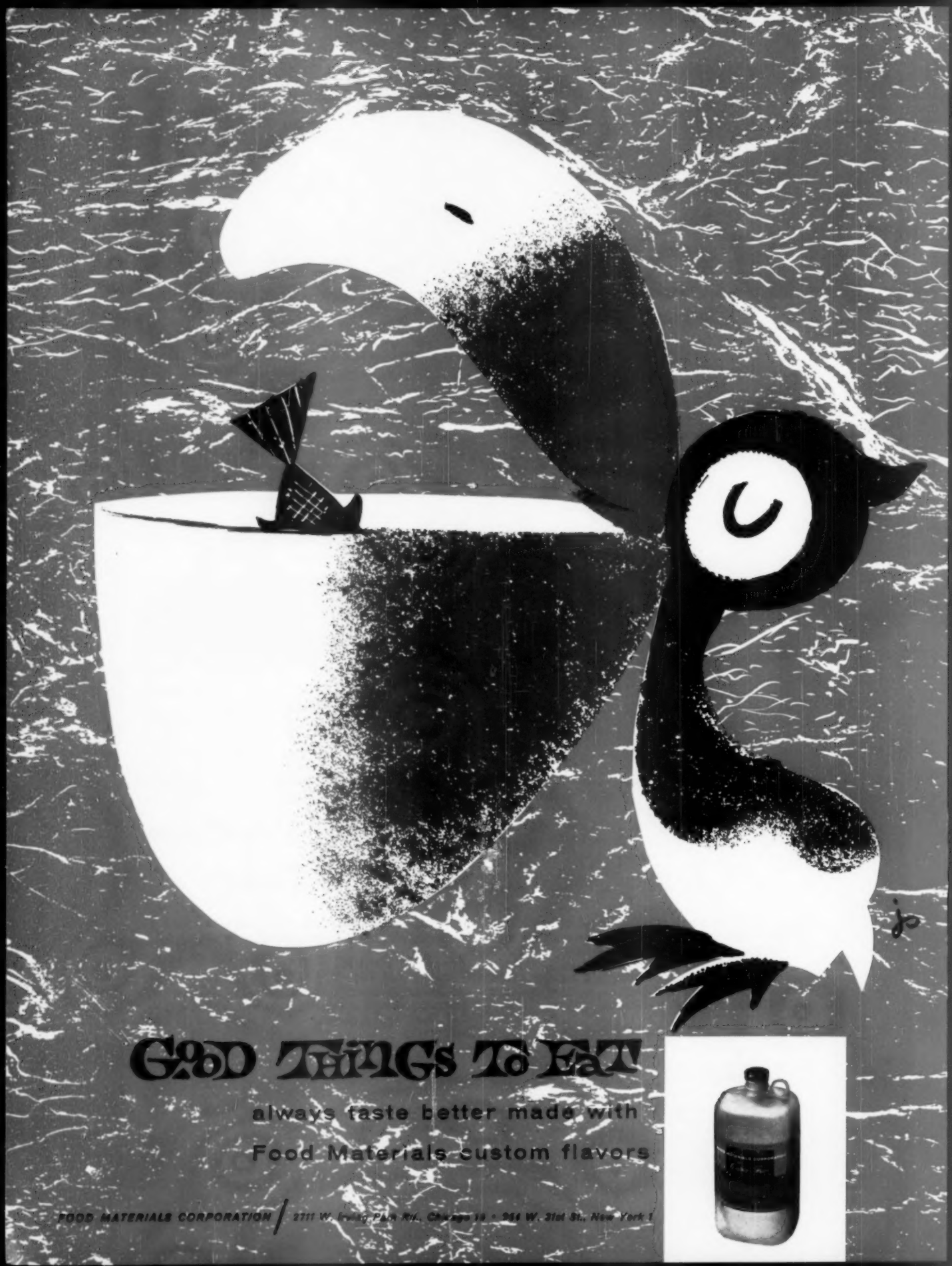
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Table 1. Ratios of residence times for fastest and slowest particles to average residence time under varied flow conditions.

Unit	Section	Method[a]	Average[b] used in ratio	Fastest particles at flow rates (lb/hr):				Slowest particles at flow rates (lb/hr):			
				1000	2000	3000	4000	1000	2000	3000	4000
A	Holding tube	C	M	.81	.81	.80		1.45	1.36	1.32	
			VT	.80	.78	.80		1.45	1.33	1.32	
	Single plate										
	no air	C	M	.45	.48	.50		1.78	1.63	1.15	
			VT	.43	.43	.50		1.69	1.47	1.15	
	with air	C	M	.32	.46			1.58	1.62		
			VT	.10	.37			.52	1.3		
	no air	MP	VT	.47	.50			1.45	1.7		
	with air	MP	VT	.19	.43			.74	1.9		
	no air	DS	VT					9.83	2.5		
A	Heating section	C	M	.66	.60	.71		1.39	1.32	1.17	
			VT	.45	.52	.70		1.04	1.15	1.16	
B	Heating section	C	M	.48	.61	.59		1.43	1.57	1.64	
			VT	.54	.65	.62		1.62	1.69	1.73	
C	Heating section	C	M		.77	.79	.79		1.08	1.15	1.14
			VT		.83	.83	.81		1.15	1.20	1.18

[a] C, conductivity; MP, motion picture; DS, dye spot.
[b] M, median; VT, volume time.

tion was flushed from flow channel with water; 3) by dye spots, calculated from time safranin remained visible in flow channel when flushed with water.

Median residence time. Calculated from time for 50% of salt to pass outlet electrode in conductivity tests.

Volume time. Average residence time calculated from flow rate and volume or cross-section area of equipment.

The sensitivity of the conductivity method was compared with the 3A Standard method (Anon., 1950) for determining the holding time in HTST pasteurizers using a Solu-Bridge Flow Timer (Industrial Instruments Inc., Jersey City, New Jersey). The 3A Standard method yielded residence times for fastest particles that were 14% longer than those obtained with the conductivity method of Watson *et al.* (1960) when applied to the holding tube of unit A, and 15% longer for the heating section of unit A. For these tests, the sensitivity of the Solu-Bridge Timer was set at 70—the highest that could be used without the timer being activated by the conductivity of the tap water.

Temperature data for use in constructing time-temperature curves were obtained in two series of experiments with each of the exchangers, with heating-water temperatures of 165 and 180°F, and thermocouples placed in the main flow channels and in side channels where channeling was expected.

All results reported are based on tests with water or dilute solutions of salt or safranin.

RESULTS AND DISCUSSION

Residence-time ratios. Residence times at selected flow rates were determined by the conductivity method for the holding tube and individual plates of unit A and the complete heating sections of units A, B, and C. To calculate selected residence-time ratios, the residence-time data were plotted, a smooth curve was drawn through the points, and the required residence times were read from the curves. Table 1 contains ratios of times for fastest and slowest particles to average residence times. For comparison, the table also includes ratios based on motion-picture results for single plates of unit A.

For the holding tube of unit A, there was excellent agreement between the ratios calculated from the median residence times and those calculated from volume times. The ratios of the residence times for the fastest particles to the volume times are higher than predicted from results of Jordan *et al.* (1952) for the low Reynolds numbers in this holding tube (4,500 to 13,500). The difference may be partly attributable to the greater turbulence in the holding tube of unit A that resulted from the use of short straight runs of pipe.

For the single plate of unit A, without air, the ratios indicate greater variability of residence times (both fast and slow particles) than for the holding tube, particularly at the lower flow rates. In the presence of air pockets in the flow channels, ratios based on volume times are misleading because the flowing liquid did not fill the flow channels.

The limited data for slowest particle times measured by observing persistence of dye in the flow channels demonstrate that this method was much more sensitive than either the conductivity or motion-picture methods as a measure of slowest particle times.

For the complete heating sections of units A and B, there is poor agreement between the median ratios and the volume time ratios at the lower flow rates. The presence of entrapped air, expected in these units at the lower flow rates, would account for these discrepancies by giving erroneous volume time values.

Comparison of ratios of fastest particle times to median times shows that residence times are more variable in units A and B than in the holding tube or unit C.

As noted, the fastest and slowest particles were arbitrarily defined in relation to the method used to detect them. The dye-spot data illustrate the limitations of the conductivity and motion-picture methods in detecting low concentrations of slow particles. Likewise, the difference between the single plate and the complete heating section of unit A in the ratio of the fastest particle to median residence times suggests a similar limitation in detecting the fastest particles. Thus, the ratio of fastest-particle residence time to median residence time could give unwarranted confidence in the safety of a process: The minimum heat treatment of a particle might be appreciably less than that calculated from this ratio.

The results contribute to understanding of the value and limitations of such criteria as "efficiency," "Gutegrad," and residence-time ratios in evaluating heating and holding equipment. Since such criteria are limited by the sensitivity of the detecting device used, they are arbitrary, empirical measures. They are helpful in indicating uniformity of residence

times, especially if the same equipment and procedure are used for their measurement. In relation to pasteurization or sterilization processes, however, they should be used with caution in predicting minimum heat treatment of individual product particles.

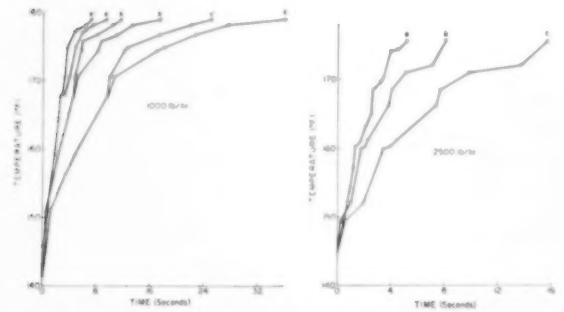

Fig. 1. Calculated rate of heating in selected flow channels in unit A (heating water, −180°F). a, fastest particles in side channel, no air; a', fastest particles in side channel, with air; b, fastest particles in center, no air; b', fastest particles in center, with air; c, slowest particles in center, no air; c', slowest particles in center, with air.

Variation in heat treatment of individual product particles. Velocity profiles determined by the motion-picture method (Watson *et al.*, 1960) were used to calculate the time required for particles to reach selected points at which temperatures were measured by thermocouples. Fig. 1 plots representative data for flow rates of 1000 and 2500 lb per hour with heating-water temperature of 180°F. To calculate possible extremes of heat treatment, it was assumed that some particles could travel in the rapidly flowing side channel or with the slowest particles in all four plate passes. On this basis, the calculated residence times at 1000 lb per hour varied from 7.2 sec for the fastest particles in the side channels with air pockets in the two down passes to 36 sec for the slowest particles through the center. At 2500 lb per hour, residence times ranged from 5.2 sec in the side channels (without air) to 15.6 sec for the slowest particles.

The intensity of the corresponding heat treatments was evaluated as described by Ball (1943) and Burton (1951). For comparison of the heat treatments with that required for pasteurization, the time-temperature relationship used was that given by a straight line drawn through and extrapolated beyond the pasteurization standards 145°F for 30 min and 161°F for 15 sec, plotted with temperature on an arithmetic scale and time on a logarithmic scale. The validity of this extrapolation has been questioned (Collins *et al.*, 1956), but it is the best available because legal standards have not been established for minimum time-temperature pasteurization treatments at higher temperatures.

Table 2 summarizes the results of the calculated intensities of heat treatments as compared with that for a minimum pasteurization treatment. At 1000 lb per hour, the minimum possible treatment for the fastest particles in the side channel was less than half that of the fastest particles in the main flow channel, and less than one-quarter that of the slowest particles. At 2500 lb per hour, differences were not as great (respectively about two-thirds and one-third).

The presence of air pockets (flow rate 1000 lb per hour) reduced the calculated heat treatment of both the fastest and slowest particles by about 40%. These calculated treatments were for fluid in the flowing streams. For food products that foam, some fluid would be present in the air pockets as foam. This fluid would be expected to have longer residence times, and hence more intense heat treatments, than that in the flowing streams.

To estimate the probability of inadequate heat treatment for some product particles, it was assumed that the side channel carried 5% of the total flow, that there was complete mixing at each crossover, and that there was no intermixing of side and center channels during flow through the plates. Under these fictitious conditions, only 0.0006% of the product would pass through the exchanger in the fastest channel in all four passes. Even this small percentage would be of concern if, in sterilization or no-hold pasteurization processes, it resulted in survival of some pathogens, spoilage organisms, or enzymes. Most of the difference between the side and center channels, however, was attributable to the last pass in which the product was at higher temperatures. If a center-channel particle flowed in the side channel for the last pass only, the intensity of the total heat treatment would have been reduced by about 25%.

Murdock *et al.* (1955) observed bacterial contamination of concentrated orange juice from the regenerative section of a plate heat exchanger. Results presented above, and also by Hansen *et al.* (1953), provide a probable explanation for build-up of thermophilic bacteria in a plate heater. Accumulation of foam in air pockets could result in long residence times for some product particles. Such pockets in flow channels maintained at a temperature that favors thermophilic growth would permit build-up of high bacterial populations and continuous contamination of product in adjacent streams. An approach to control of the contamination, in addition to those suggested by Murdock *et al.* (1955), would be to increase product flow rate enough to avoid accumulation of air (foam) in downflow passes. If the increased flow rate caused too much decrease in the heat-transfer capacity of the equipment, the plate arrangement

Table 2. Comparison of possible extremes of heat treatment of individual particles during passage through heating section of unit A.

Flow condition	Treatment ratio[a]	
	Flow rate (1000 lb/hr[b])	Flow rate (2500 lb/hr[c])
Fastest particle in side channel	26.8	5.17
Fastest particle through center, no air	55.7	8.02
Fastest particle through center, with air	32.7	
Slowest particle, no air	119.0	16.05
Slowest particle, with air	69.8	

[a] Ratio of calculated treatment of particle to minimum pasteurization treatment.
[b] Maximum temperature 178.8°F.
[c] Maximum temperature 175.6°F.

could be changed by reducing the number of channels in the downflow passes without changing the upflow passes. Under some conditions this modification would eliminate air entrapment with little change in pressure drop or heat-transfer effectiveness.

In many HTST pasteurizers used for milk, conditions in the raw-milk side of the regenerative section are frequently conducive to air-pocket formation. A common public health requirement is that this section of the exchanger be operated under negative pressure. To minimize difficulties with vapor lock in the timing pump, a low pressure drop in the regenerative section is necessary, and is sometimes achieved by dividing the flow among more plates than are used in other sections of the exchanger. The resulting low flow rate favors air entrapment in downflow passes.

Several observations from this study warrant special emphasis:

1) Measured variations in heat treatment of individual particles in a heat exchanger depend to a large extent on the sensitivity of the method used for detecting the fastest and slowest particles.

2) Heat treatment applied to individual particles of product passing through plate heat exchangers may vary appreciably. Two methods readily showed threefold variations, but much greater variation was indicated in a small proportion of the fluid by dye-spot data.

3) The terms fastest and slowest as used in studies in this field are arbitrary, and may give the processor or public health official a false sense of security, particularly in relation to heat treatment of the fastest particles.

4) Zones of low velocity (including air pockets) may contribute to several undesirable conditions, depending on the product and the processing conditions, such as fouling of the heat-transfer surface, build-up of thermophilic organisms, and increase in cooked, scorched or other off flavors.

5) Greater attention should be given to avoiding air pockets in down-flow passes and in crossovers of plate heat exchangers. Factors to be considered include design of the plate, area of crossover ports, arrangement of plates in the exchanger, fluid flow rates, and probably the physical properties of the fluid.

ACKNOWLEDGMENT

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Cloud Loss in Bottled Carbonated Orange Beverages Induced by a Soluble Solids Concentration Gradient

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(Manuscript received January 19, 1961)

SUMMARY

Bottled cloudy-type beverages containing citrus juice sometimes separate into well defined layers of differing cloud density in a form of cloud loss distinct from enzymatic or nonenzymatic flocculation. A direct cause was found to be incomplete mixing of the bottling sirup and carbonated water. Successive layers are shown to differ in soluble solids concentration, but the magnitude of this difference is relatively much less than the difference in cloud density. It was demonstrated that a soluble solids gradient causes otherwise-stable cloud particles to gravitate toward the more concentrated strata. A possible mechanism is suggested.

MOST REPORTED WORK on the stability of suspended cloud in citrus juices is concerned with the effect of pectic enzymes. Cloud stability problems can exist, however, in citrus beverages containing juices adequately heat-treated to inactivate enzymes (Stevens, 1940; Stevens and Pritchett, 1952). In such cases it is possible to confirm the effectiveness of the heat treatment by testing for residual enzyme activity (Stevens, 1941).

The products most likely to be affected by nonenzymatic cloud loss are cloudy-type carbonated beverages and bottled lemon juice. Since these are usually packaged in clear-glass bottles, any variations in cloud density are readily apparent.

Cloud in citrus juices is made up of finely divided pulp particles, natural pigment particles, and, if flavoring oils have been added, oil emulsion droplets. The last two types of cloud particles are small enough to be subject to Brownian movement, which helps to keep them in suspension. Pulp particles generally have a tendency to settle, the rate depending on particle size, convection currents, and other factors not completely understood. At times, pulp particles tend to agglomerate, then settle out rapidly, carrying down the other components of the cloud. A remedy for one type of settling of this nature was the subject of patents by Stevens and Pritchett (1952, 1956). Nonenzymatic cloud loss, however, is not always typified by rapid general clearing throughout the beverage. With essentially pure bottled lemon juice, it is sometimes observed that only a portion of the bottle becomes clear. It may be hypothesized that the natural pectin in the juice forms a loose gel, which by a syneresis-like process squeezes out a clear liquid. Such cloud loss is typified by a cone-shaped structure of cloud particles within the bottle, similar in shape to the neck of the bottle. The intact cone can be moved about by gentle agitation of the bottle, but more severe agitation redisperses the cloud.

Beverages containing about 5% juice, in which the possibility of gel formation is remote because of dilution, may also exhibit a layering effect. Still air and constant temperature, which eliminate convection currents, favor this type of cloud separation. In some instances sedimentation, combined with the effect of viewing a thinner layer of beverage in the narrow neck of the bottle, gives a false impression of a layering effect.

In other cases, however, an actual separation into well-defined layers does occur. Beverages showing this effect are usually prepared by first placing a flavored sirup in bottles, then filling the bottles with carbonated water, followed by capping and a mixing procedure. Incomplete mixing of the contents of the bottles could thus be a contributory factor. However, the bottles are often observed as initially having uniform cloud distribution; the separation into layers occurs only after three to five days. More than two layers may appear. Thorough agitation redisperses the cloud, and separation does not recur on further standing.

This last type of cloud loss has been given the name "neck cloud loss" (NCL), referring to the neck of the bottles. Work on the cause of NCL is reported here.

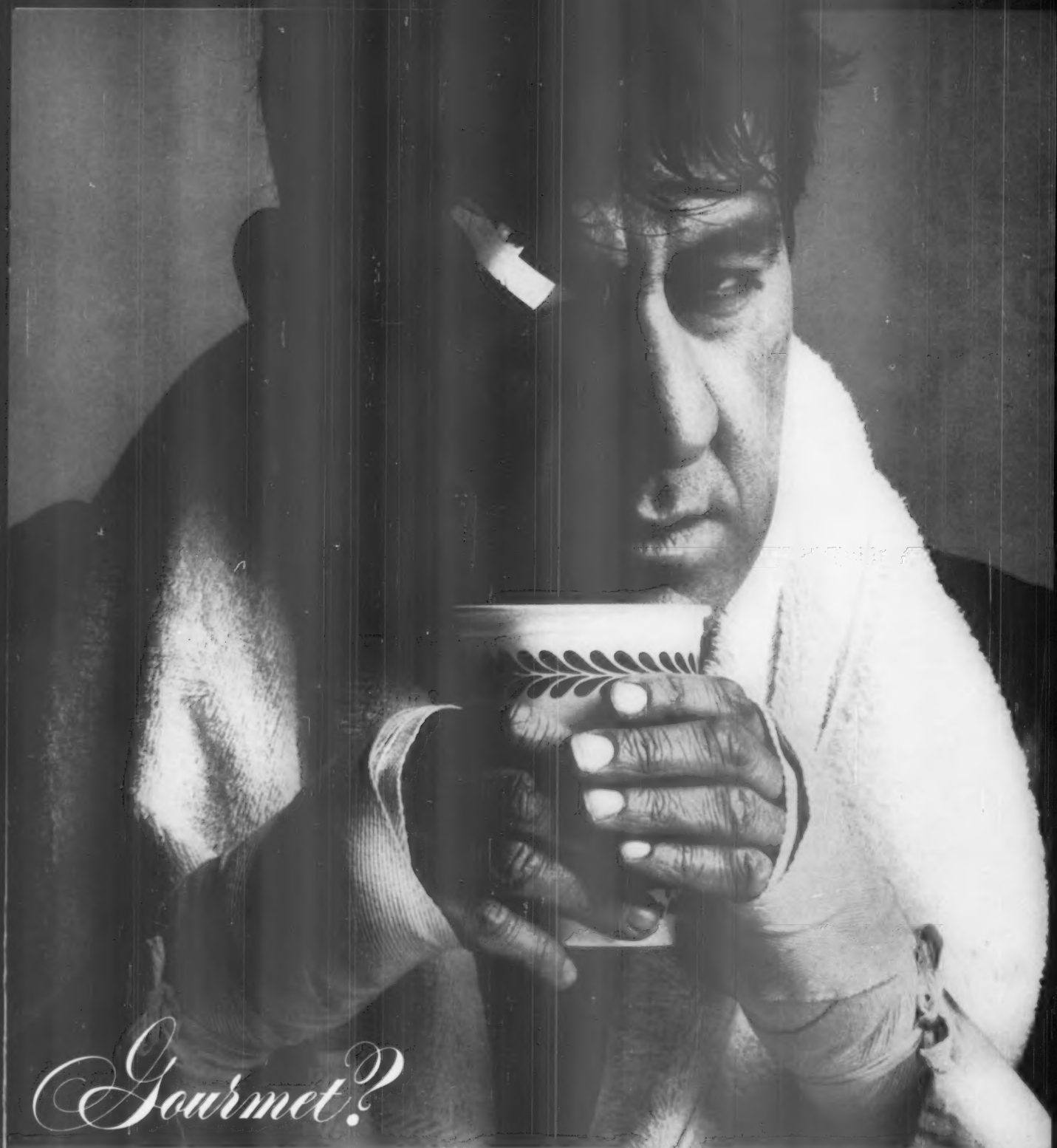
PROCEDURE

Carbonated bottled beverages. Pasteurized orange juice, concentrated to 65° Brix, was compounded into a typical beverage base, containing (besides the orange juice) citric acid, salt, sodium benzoate, sodium citrate, water, and a flavoring-oil emulsion. The emulsion, prepared separately, contained orange oil, lemon oil, brominated vegetable oil, a gum emulsifier, and water.

For use, 50 ml (62.2 g) of the above base were mixed with 850 ml (1089.0 g) of 32° Be sugar sirup, and 42.3 ml of the resulting sirup was placed in each clear 8-oz carbonated-beverage bottle. The bottles containing the sirup were chilled to about 35°F, and then filled with cold carbonated water. The water was flowed gently down the sides of the bottles to avoid foaming and mixing of the water and sirup. It was necessary to minimize mixing during filling so that later, after the bottles were crown-capped, a controlled degree of mixing could be obtained by gently inverting each bottle the desired number of times.

Normal composition of the beverage is 12.8% soluble solids, 0.130% citric acid and 0.02% sodium benzoate.

Noncarbonated bottled beverages. The base described above was prepared in the same way except that sufficient sodium benzoate was added to the sirup to increase the amount in the



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beverage to 0.05%. Extra citric acid was also added to maintain the normal pH existing before the sodium benzoate addition. Vacuum-deaerated chilled water was used to fill the bottles in the same manner as for the carbonated samples.

Noncarbonated beverages in test tubes. In addition to the beverage base already described, two simulated bases were prepared, one with no flavoring oils and one with no juice. In the first case water was substituted for the emulsion, and in the second sugar, citric acid, sodium citrate, and water were substituted for the orange juice. These three bases were made directly into beverages by mixing with dry sugar and water. Additional sodium benzoate was included to give a total of 0.15% in the beverages, and sufficient citric acid was added to maintain the normal pH.

Two beverages were made from equal amounts of each base, one containing 11.0% soluble solids and the other 13.0% soluble solids. The beverages were placed in 22 × 200-mm test tubes, which were closed with rubber stoppers.

Storage of samples. Beverages were stored for observation in a room held at 28°C. Temperature fluctuation was very slight, but there was some air movement whenever the forced-air heater was operating.

Analytical methods. Bottled samples were observed in a darkened room by means of a light directed up through the bottom of the bottle. The light was focused with lenses so that no convection currents would be created in the beverage by heat transfer from the light bulb. Regions of more dense cloud diffused the light to a greater extent and appeared brighter than regions of less dense cloud. Fairly small differences in cloud density could thus be perceived. Two degrees of cloud loss were recognized: complete clearing in which the region observed contained virtually no cloud, and partial clearing for any cloudy region that appeared as a distinct layer having less cloud than the layer immediately below.

Because of the light-diffusing effect of the curved bottom of the test tubes, visual observations could not be accurate. Instead, two 10-ml samples were carefully withdrawn by pipette, one from about 1 in. below the surface of the liquid, the other from 1 in. above the bottom of the tube, and the relative cloud density of these samples was read on a Klett-Summerson Photoelectric Colorimeter, zeroed on water, using a No. 66, red filter.

Brix. Samples were withdrawn from visible layers in bottled beverages by pipette. Readings with a Zeiss refractometer were made on these samples and on samples removed from the test tubes. Two to three readings were made on each sample, and the average reading, corrected to 20°C is reported. These values are used as a measure of soluble solids concentration.

RESULTS

Effect of mixing. Agitation of the bottles after NCL has appeared redisperses the cloud and the NCL does not recur, suggesting that the extent of mixing after bottling may be an influential factor. To determine if that is so, an experiment was prepared comprising four groups of 8 bottles each. After capping, the first group was given two inversions, the second group four inversions, the third group six inversions, and the last group eight inversions. After mixing, all bottles appeared to have uniform cloud density.

Table 1 gives the number of bottles in each group showing complete, partial, or no NCL after 14 days. It is evident that even eight inversions of the bottles does not eliminate NCL completely.

Table 1. Effect of amount of mixing on incidence and severity of neck cloud loss in a carbonated orange beverage.

Amount of mixing—number of times each bottle inverted	Total bottles	Complete clearing	Partial clearing	No clearing
Bottles stored 3 days at 28°C				
two	8	7	1	0
four	8	7	0	1
six	8	1	3	4
eight	8	2	1	5
Bottles stored 14 days at 28°C				
two	8	6	0	2
four	8	1	5	2
six	8	0	3	5
eight	8	0	3	5

Relation of soluble solids distribution to NCL. Although incomplete mixing is apparently a cause of NCL, it is unlikely that after several inversions of a bottle there should be such an uneven distribution of the sirup that the regions having almost no cloud should also be nearly free of soluble solids. To determine the relation, if any, between soluble solids distribution and NCL, a series of five bottles of beverage was prepared and each bottle given two inversions. Still or plain water, rather than carbonated water, was used so that the bottles could be opened for sampling without the mixing that results from carbon dioxide evolution. Visual examinations were made, and bottles were opened for withdrawal of samples at intervals up to 16 days. Table 2 shows the depth of layers, degree of cloud loss, and ° Brix of each layer.

Definite differences in soluble solids concentration are associated with each layer. However, there is no direct quantitative relationship between differences in soluble solids concentration and degree of NCL. For example, the greatest difference observed within a bottle, that of 1° Brix for sample C, is of far less relative magnitude than the difference in cloud density between the corresponding layers. This suggests that more than incomplete mixing is involved in NCL.

The degree of difference in soluble solids concentration between layers does not appear to change with time, indicating that diffusion and convection contribute very little to soluble solids equalization.

Effect of a soluble solids gradient. It can be reasoned that the layers of differing soluble solids concentration within the bottles are established fairly soon after mixing. Probably, volumes of liquid more concentrated than the solution surrounding them settle rapidly until they arrive at a region matching their own specific gravity. If this were a lengthy process, mixing would tend to be complete. NCL, however, does not usually appear for at least two days, and sometimes requires as long as five days. Also, regions of partial clearing may eventually become completely clear, indicating a continuing process. A possible explanation is that the soluble solids gradient in itself destabilizes the cloud particles, causing them to settle.

To test this hypothesis, an 11° Brix beverage was layered in test tubes on top of an equal volume of a 13° Brix beverage. For control purposes both beverages were also placed in test tubes individually. Beverages from the two simulated bases, one made with no juice and the other with no flavoring oil, were similarly prepared. Table 3 shows observations of cloud density

Table 2. Relationship between neck cloud loss and soluble solids distribution in a noncarbonated orange beverage.

Bottle															
A (5 days)			B (7 days)			C (9 days)			D (11 days)			E (16 days)			
Depth of layer	Extent of NCL	° Brix	Depth of layer	Extent of NCL	° Brix	Depth of layer	Extent of NCL	° Brix	Depth of layer	Extent of NCL	° Brix	Depth of layer	Extent of NCL	° Brix	
(in.)			(in.)			(in.)			(in.)			(in.)			
1¼	complete	12.2	5	partial	12.6	1⅝	complete	12.3	2¼	complete	12.6	2¼	complete	12.6	
2¼	partial	12.3	6	none	13.2	3½	partial	12.7	6	none	13.1	5	partial	12.7	
4	partial	12.6				6	none	13.3				6	none	13.0	
6	none	12.8													

Table 3. Showing the soluble solids gradient-induced migration of beverage cloud particles to the region of highest soluble solids concentration.

Beverage placed in tube	Sample from top of tube		Sample from bottom of tube	
	Observed ° Brix	Cloud density	Observed ° Brix	Cloud density
Complete[a]				
11° Brix	11.0	71	11.0	72
13° Brix	13.0	74	13.0	74
11° Brix over 13° Brix	11.0	47	13.0	95
Without flavoring oil[b]				
11° Brix	10.8	36	10.9	36
13° Brix	13.0	36	13.0	36
11° Brix over 13° Brix	10.9	26	12.8	40
Without orange juice[b]				
11° Brix	10.9	30	10.9	32
13° Brix	13.0	31	13.0	32
11° Brix over 13° Brix	11.0	24	12.9	41

[a] Tabulated data are average values for four tubes after 8 days at 28°C.

[b] Tabulated data are average values for two tubes after 9 days at 28°C.

and ° Brix of the complete beverages after 8 days, and of the simulated beverages after 9 days.

All three beverages, regardless of soluble solids content, were stable by themselves during observation. It is apparent, however, that a soluble solids gradient induces a definite migration of cloud particles from the regions of lower soluble solids content to the regions of higher soluble solids concentration. Both the cloud derived from juice and that derived from the flavoring oil emulsion are affected. This is not a flocculation phenomenon, since the cloud transferred to the lower phase is stable there.

DISCUSSION

Nothing in the data suggests why a soluble solids concentration gradient should cause a migration of cloud particles. Simple sedimentation is not an adequate explanation since the beverages of uniform soluble solids concentration in Table 3 showed no tendency to decrease in cloud density at the top or increase in cloud density at the bottom of the tubes. Unfortunately, the complex system comprising the cloud in orange-juice-containing beverages does not lend itself to study of the factors that may affect the stability of each of the various types of particles present.

Two recent publications, however, may offer some explanation for the behavior demonstrated. Sterling and Chichester (1960), studying adsorption of glucose by cooked peach tissue, concluded that the cell walls of such tissue attract glucose. As a result, a gradient of decreasing concentration toward the center of the cell is established through the movement of glucose molecules toward the cell wall. To apply this finding to NCL, it is necessary to assume that sucrose is attracted to orange cell walls in the same manner as glucose is attracted to peach tissue. Then, in contrast to the situation in intact peach tissue, the cell wall fragments in orange beverages are relatively free to move and the sugar concentration gradient is apparently fixed, as indicated by the persistence of soluble solids gradients even after 16 days (Table 2). It follows that a small mobile pulp particle in such a concentration gradient would be attracted toward the greater concentration.

Supplementing this reasoning is the finding by Nicholas *et al.* (1960) that convection currents are confined within layers in a system composed of layers of less concentrated solutions on top of layers of similar, but more concentrated, solutions. Thus, in a beverage, convection would tend to maintain a uniform distribution of cloud particles within each layer of uniform soluble solids concentration. Particles coming in the vicinity of a soluble solids gradient at the lower boundary of a layer, however, would tend to migrate toward the higher soluble solids concentration. This migration would be influenced both by the attraction of the more concentrated solution for the cloud particles and by sedimentation. The greater effective specific gravity of particles in equilibrium with the more concentrated solution would reduce any tendency for migration to the less concentrated region. The net result is transfer of cloud to the more concentrated region. In the case of multiple layers, each successive layer from the top down would become clear as gain from the layer immediately above failed to compensate for loss to the layer immediately below.

An explanation is thus offered for the manner in which incomplete mixing causes NCL. The observations attach new significance to thorough mixing in the practice of bottling cloudy beverages.

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Studies in Canning Processes. IV.

Lag Factors and Slopes of Tangents to Heat Penetration Curves for Canned Foods Heating by Conduction

(Manuscript received August 10, 1960)

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SUMMARY

The lag factor j and slope f of the asymptote to heat penetration curves are important parameters in process calculations. Since in practice the asymptote is not known these parameters are obtained from a tangent drawn to the heating curve. The values of these parameters for the tangent may differ appreciably from those of the asymptote, the difference depending on the ratio of can height to can diameter and the position on the heating curve at which the tangent is drawn. Curves are given showing these effects.

HEAT PENETRATION data on canned foods are generally displayed graphically by plotting a dimensionless temperature on a logarithmic scale against time on a linear scale; the dimensionless temperature normally used is the ratio of the temperature difference between the retort and the can center at any time to the value of this temperature difference at the beginning of the process. Theoretically, for conduction heating of products, the resulting heating curves approach a straight-line asymptote. The lag factor j and the slope[a] f of the asymptote are important parameters in the calculation of canning processes (Ball and Olson, 1957).

In practice the length of heating curve that can be used to evaluate j and f is frequently limited. When the retort temperature is high, the process time required for sterilizing the pack is often short. But even when processing is continued until the center temperature is nearly equal to the retort temperature, the nature of the logarithmic plot of the heating curve means that even small errors in temperature measurements cause the later points to be widely scattered, making the curve ill-defined in that region. Variation in the retort temperature, due to drift in the controller or a fluctuating steam supply, increases this inaccuracy and sometimes results in a pronounced alteration in the slope of the curve near the end of the heating phase.

Since the exact position of the asymptote to the heating curve is not known, a tangent is drawn to the experimental curve and the parameters j and f of this tangent are used in calculating the lethal value of the process. This tangent is drawn at some point after the curve first appears to become linear but before the irregular section near the end of the process.

[a] f is not strictly the slope of the curve, but it is customary to call it that.

The values of the parameters for the tangent sometimes differ markedly from those for the asymptote. It is not possible to estimate these differences for every practical case, because the processing conditions are often quite complex. It is possible, however, to estimate them for a somewhat simplified case, which nevertheless is reasonably close to most practical conditions. The simplifications assumed are:

1) the retort reaches its operating temperature instantaneously,

2) the temperature in the can is uniform at the beginning of the process,

3) the product heats purely by conduction,

4) the thermal properties of the product do not vary with the temperature,

5) the headspace offers no resistance to heat transfer, and

6) the heat transfer coefficient from the heating medium to the outside surfaces of the can is infinite.

The effects of taking tangents to heating curves seem to have been investigated first by Olson and Jackson (1942). They made the simplifying assumptions about the process that are listed above, and quoted the exact solution for the temperature distribution in the can. From this they derived an approximate formula for the j value of a tangent, and gave curves of j values calculated from this approximate formula; they did not consider the effect on f.

Because the accuracy of their curves is open to question and because these were recently reproduced in a standard work on canning processes by Ball and Olson (1957), it was thought appropriate that more accurate data be made available.

The exact formulae used in the present calculations are given in the appendix. Following Olson and Jackson (1942), the effects of taking tangents are expressed in terms of two parameters: p, which takes into account the position on the heating curve at which the tangent is drawn; and q, which takes into account the shape of the can. These are defined as:

p = number of log cycles from the beginning of the process to a point on the asymptote having the same abscissa as the point on the heating curve at which the tangent is drawn, and

$q = (L^2/a^2)$, in which 2L is the height of the can and 2a the diameter.

Fig. 1 shows the results obtained for the lag factor

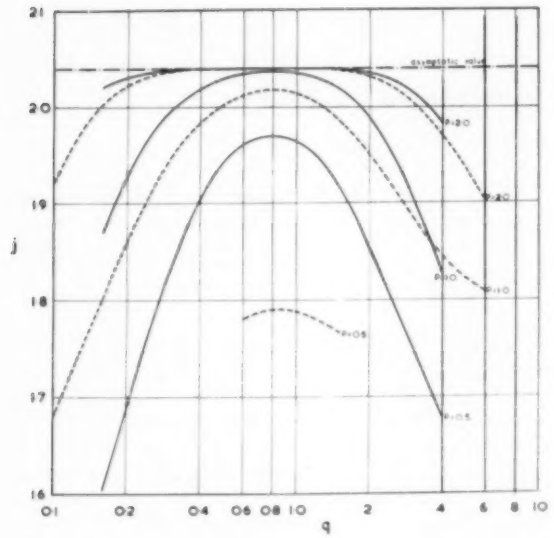

Fig. 1. The lag factor j as a function of q, with p as parameter. ----, formula given by Olson and Jackson (1942). ————, present results.

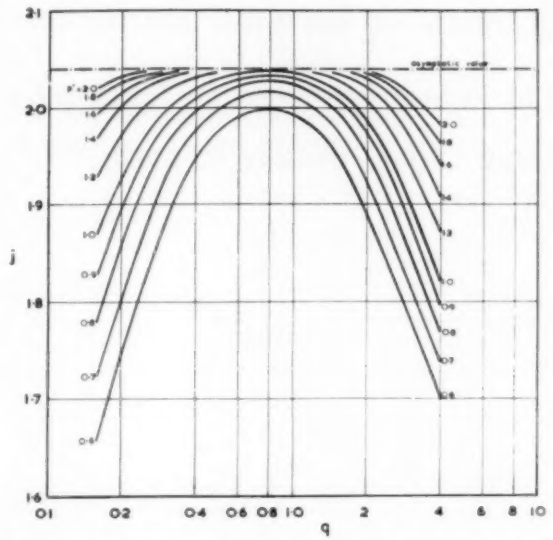

Fig. 2. The lag factor j as a function of q, with p′ as parameter.

j, together with those given by the earlier authors. Only three values of the parameter p are given for comparison, since the results are believed to be of more practical use when displayed in a slightly different way. The curves given by Olson and Jackson are seen to be of the right general shape but considerably overestimate the effects of taking tangents in place of asymptotes.

The parameter p is clearly defined in terms of the asymptote; although this is convenient mathematically it is not so useful in practice, since, after a thermal process, the heating curve is known but the true position of the asymptote is not. The effects on j are therefore expressed more fully in Fig 2, in which the parameter p is replaced by p′, defined as:

p′ = number of log cycles from the beginning of the process to the point on the heating curve where the tangent is drawn.

It is clearly not necessary to know the position of the asymptote to know p′.

In this figure the interval in p′ is small enough for rapid interpolation when necessary. The smallest value of p′ chosen is 0.6 since it would rarely have a value smaller than this in practice. It is interesting to note that the maximum value of j does not occur at q = 1.0, but near q = 0.8.

The value of the slope f of the tangent will also be different from that of the asymptote. The present calculations have been extended to include the effect on f, and the results are displayed in Fig. 3. The quantity Δf is defined as:

Δf = the difference between the f values of the tangent and the asymptote expressed as a percentage of the f value of the asymptote.

In Fig. 3 the abscissa q and the parameter p′ are the same as in Fig. 2.

The effects on f of taking tangents instead of asymptotes are seen to be similar to those on j; when expressed as a percentage, however, f is affected much less than is j.

It is important to note finally that experimental values of j and f can be affected as much by departures from the simplifying assumptions listed earlier as by the use of tangents in place of asymptotes. For example, the thermal resistance of the headspace could decrease the value of j by up to 10%, depending on the size and shape of the can, while the increase in thermal diffusivity with temperature increases the value of j. Some of these other factors were discussed more fully in the second paper in the present series (Evans, 1958).

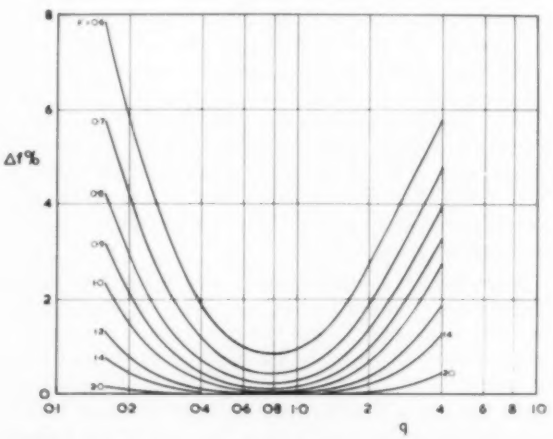

Fig. 3. Δf as a function of q, with p′ as parameter.

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APPENDIX

Mathematical procedures

When the simple processing conditions given in the text are satisfied, using a formula given by Carslaw and Jaeger (1959, p. 225), the temperature at any point in the can may be expressed in the following dimensionless form:

$$\frac{T - T_1}{T_0 - T_1} = \frac{8}{\pi} \sum_{n=0}^{\infty} \sum_{m=1}^{\infty} \frac{(-1)^n}{(2n+1)} \cdot \cos (2n+1) \frac{\pi x}{2L} \cdot \frac{J_0 (\alpha_m r/a)}{\alpha_m J_1 (\alpha_m)} \cdot \exp - Kt \left\{ \frac{(2n+1)^2 \pi^2}{4 L^2} + \frac{\alpha_m^2}{a^2} \right\} \quad [1]$$

in which:

T	=	temperature at time t of the point in the can specified by the coordinates x and r,
T_1	=	retort temperature,
T_0	=	initial temperature of the product,
x	=	distance of the point whose temperature is being specified from the plane drawn through the center of the can and parallel to its ends,
r	=	distance of the point being specified from the central axis of the can,
2L	=	height of can,
2a	=	diameter of can,
K	=	thermal diffusivity of the product,
α_m	=	roots of $J_0 (\alpha) = 0$,
J_0, J_1	=	Bessel functions of the first kind (Carslaw and Jaeger, 1959, Appendix III), and
n,m		take integral values and specify the term in the series.

At the center of the can, where x = r = 0, equation 1 simplifies since the cosine term and the Bessel function occurring in the numerator are both unity. If we abbreviate by writing:

$$A_{n,m} = \frac{8}{\pi} \frac{(-1)^n}{(2n+1)} \frac{1}{\alpha_m J_1 (\alpha_m)} \quad [2]$$

and

$$B_{n,m} = \left\{ \frac{(2n+1)^2 \pi^2}{4L^2} + \frac{\alpha_m^2}{a^2} \right\} \quad [3]$$

then for the center temperature, T_c, equation 1 takes the form

$$\frac{T_c - T_1}{T_0 - T_1} = \sum_{n=0}^{\infty} \sum_{m=1}^{\infty} A_{n,m} \exp \left\{ -B_{n,m} Kt \right\} \quad [4]$$

It should be noted that a double series such as that given in equation 1 can be evaluated numerically either as a product of two individual series or as a single series whose terms are in the form of products. The latter method was used in the present work, and equation 4 is written in this way, the two summation signs being retained since summation still takes place over both n and m.

Heat penetration curves are usually plotted with a logarithmic scale on the temperature axis. The asymptote to this curve is the straight line represented by the first term in equation 4, namely, when n = 0 and m = 1. A point on this asymptote p log cycles from the beginning of the process will occur at a time that we denote by t_p.

The equation to the tangent at a point on the heating curve with the same abscissa t_p can be obtained from equation 4. Its slope is obtained by taking logarithms of equation 4 and differentiating with respect to t, and its intercept on the temperature axis is log j_p, in which j_p is the lag factor appropriate to this tangent. Rearranging this equation gives for log j_p the expression:

$$\log j_p = \log \left\{ \sum_{n=0}^{\infty} \sum_{m=1}^{\infty} A_{n,m} \exp (-B_{n,m} Kt_p) \right\} + \frac{t_p \sum_{n=0}^{\infty} \sum_{m=1}^{\infty} K A_{n,m} B_{n,m} \exp (-B_{n,m} Kt_p)}{2.303 \sum_{n=0}^{\infty} \sum_{m=1}^{\infty} A_{n,m} \exp (-B_{n,m} Kt_p)} \quad [5]$$

in which the numerical factor 2.303 serves to convert from natural to common logarithms.

For the asymptote only one term in each of the series in equation 5 is retained, and the equation is:

$$\log j_a = -p + t_p \frac{K B_{0,1}}{2.303} \quad [6]$$

in which:

j_a = lag factor of the asymptote which has the value 2.0397, and

$B_{0,1}$ is the first of the functions $B_{n,m}$ defined in equation 3.

The negative sign preceding p arises from the fact that $(T_c - T_1)/(T_0 - T_1)$ in equation 4 is less than unity.

From equation 6, t_p can be written as:

$$t_p = \frac{2.303}{K B_{0,1}} \left\{ \log j_a + p \right\} \quad [7]$$

which, when substituted into equation 5, gives:

$$\log j_p = \log \left(\sum_{n=0}^{\infty} \sum_{m=1}^{\infty} A_{n,m} e^{-C_{n,m}} \right) + \frac{\sum_{n=0}^{\infty} \sum_{m=1}^{\infty} A_{n,m} C_{n,m} e^{-C_{n,m}}}{2.303 \sum_{n=0}^{\infty} \sum_{m=1}^{\infty} A_{n,m} e^{-C_{n,m}}} \quad [8]$$

in which

$$C_{n,m} = 2.303 (\log j_a + p) \frac{B_{n,m}}{B_{0,1}} \quad [9]$$

in which

$$\frac{B_{n,m}}{B_{0,1}} = \frac{\left(\frac{(2n+1)^2 \pi^2}{4q} + \alpha_m^2 \right)}{\left(\frac{\pi^2}{4q} + \alpha_1^2 \right)} \quad [10]$$

and α_1 has the value 2.4048.

Examination of equation 8, together with equations 9 and 10, shows that log j_p is a function of the two parameters p and q only. Calculations of j_p for various values of p and q were made using equation 8.

To express the results in terms of p′ instead of p is straightforward since the first quantity on the right hand side of equation 8 is p′.

The respective "slopes" of the tangent and the asymptote can be obtained from equations 5 and 6. From these the quantity Δf, defined in the text, is expressed in terms of the parameters p and q as:

$$\Delta f = 100 \left\{ \frac{\left(\sum_{n=0}^{\infty} \sum_{m=1}^{\infty} A_{n,m} e^{-C_{n,m}} \right)}{\left(\sum_{n=0}^{\infty} \sum_{m=1}^{\infty} A_{n,m} \frac{B_{n,m}}{B_{0,1}} e^{-C_{n,m}} \right)} - 1 \right\} \quad [11]$$

where $A_{n,m}$ is defined in equation 2, $B_{n,m}$ in equation 10, and $C_{n,m}$ in equation 9.

A Technical Note

Effectiveness of Ascorbic Acid in Preventing the Darkening of Oil-blanched French-fried Potatoes[1]

Some potatoes turn dark or develop dark spots after they are cooked. This has become a problem with oil-blanched French-fried potatoes (Hawkins *et al.*, 1959), an important product developed in the last few years. In susceptible potatoes the reaction can be prevented by treatment with certain chemical compounds (Hawkins *et al.*, 1959; Smith, 1958; Smith and Kelly, 1944, 1945). The use of these compounds on a commercial scale, however, might create disposal problems and possible toxicity. We thought a possible way of avoiding this problem might be the use of ascorbic acid. Records (Antle and Bohn, 1953; Greig and Smith, 1960; Johnson and Guadagni, 1949; Olson and Treadway, 1949) of using ascorbic acid for this or similar purposes give insufficient data for comparing its effectiveness with that of other compounds, or for assessing its applicability to oil-blanched French-fried potatoes.

We found that ascorbic acid will keep susceptible potatoes from darkening when they are cooked by boiling or put through the oil-blanched French-fried process. This latter was carried out as previously described (Hawkins *et al.*, 1959), with the ascorbic acid applied during the blanching in water before partial cooking in oil.

In one lot of potatoes the minimum effective concentration of ascorbic acid was 0.5% for boiling, and 1% for preliminary blanching of the lightly fried product.

A second lot of potatoes did not develop the dark color so readily, but if the lightly fried product was blanched for 3 min during preparation, the degree of darkening in the finished product was about the same as with the first lot after the regular blanching period of 5 min. Oil-blanched French-fried potatoes were prepared from tubers of this lot with a number of chemical substances added to the blanching water in various concentrations. The results are shown in Fig. 1. The concentrations of solutions noted there were the lowest that prevented the darkening.

Ascorbic acid by itself, therefore, is effective in preventing after-cooking darkening in potatoes, but the amount necessary is relatively large. With oil-blanched French-fried potatoes, it is comparable in this respect to citric acid (Hawkins *et al.*, 1959) and sodium acid pyrophosphate. In contrast, some of the salts of ethylenediaminetetraacetic acid (EDTA) are effective in considerably smaller amounts. It may be expected that the effective amount of any such agent will vary according to the concentration of chromogenic materials in the potato and the conditions of cooking.

Fig. 1. The effect of various chemicals on darkening of oil-blanched French-fried potatoes. This is the appearance 2 hr after completion of the process. Blanching was done in: 1) untreated water; 2) 0.3% disodium EDTA; 3) 1% sodium acid pyrophosphate; 4) 1% ascorbic acid; 5) 0.5% calcium disodium EDTA.

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(Continued from page 28 preceding research section)

min A, thiamine, and ascorbic acid content of a few dried fruits. These data, from the U. S. Department of Agriculture's Home and Garden Bulletin No. 72 (September 1960), are recalculated in terms of amounts per 100 g of dried fruit.

	Amounts per 100 grams of dried fruit		
Dried fruit	Vitamin A (International units)	Thiamine (mg)	Ascorbic acid (mg)
Apricots	10,926	0.013	12.6
Peaches	3,900	0.012	17.5
Prunes	1,340	0.06	3.0
Raisins	18.7	0.11	1.2

This table clearly shows that dried fruits are not an important source of thiamine but contribute substantial amounts of vitamin A and ascorbic acid. These data assume even greater importance in view of the fact that sulfur dioxide treatment of dried fruits protects the vitamin A and ascorbic acid but destroys the minimal amounts of thiamine.

2) In evaluating the significance of the data reported by Fitzhugh, Knudsen, and Nelson as related to the human diet, it must be remembered that those investigators studied the effects of varying amounts of sodium bisulfite in the total diet of the experimental animals. The amounts found to be deleterious could not possibly be approached in the human diet in which SO_2-treated dried fruits represent a small part of the total diet. Moreover, the addition of sodium bisulfite to the basal diet of experimental animals permits little or no loss of SO_2. Under the acid conditions existing in dried fruit, loss of SO_2 is appreciable, resulting in less SO_2 at time of consumption, especially after cooking, than existed at time of application. Large quantities of dried fruit can be fed to rats, and it would have been more pertinent to the problem at hand to have compared the effects of feeding SO_2-treated and untreated dried fruits. A. F. Morgan and A. Field (*J. Biol. Chem.* **82**, 579–586; 1929) demonstrated that feeding unsulfured dried peaches to guinea pigs resulted in severe scurvy whereas control animals on sulfured dried peaches were free of scurvy.

3) The deteriorative changes occurring naturally in unsulfured dried fruit, even in the absence of microbial spoilage, not only have unfavorable effects on flavor and texture but may be potentially toxic. For example, the well-known Maillard reaction, responsible for the browning discoloration, involves combination of reducing sugars with the amino groups of amino acids, peptides, and proteins. It is not known whether these primary amine compounds are toxic. However, it has recently been shown that a comparable reaction between hexose and a secondary amine, such as dimethylamine, produces a compound that is toxic in small doses, both acutely and chronically. Studies of such compounds have been reported by A. M. Ambrose, D. J. Robbins, and F. DeEds (*Proc. Soc. Exptl. Biol. Med.* **106**, 656; 1961) and by W. Cutting, A. Furst, D. Read, G. Read, and H. Parkman (*Proc. Soc. Exptl. Biol. Med.* **104**, 381; 1960).

In summary, it can be said that sulfured dried fruit is better than unsulfured from a nutritional point of view because of preservation of vitamins A and C, prevention of deteriorative changes and microbial spoilage, and avoidance of Maillard reaction products, which may or may not be toxic. Before toxic levels of sulfur dioxide can be reached in sulfured dried fruits these products would be unacceptable because of the disagreeable taste produced by high levels of SO_2.

Finally, it should be emphasized that evidence of thiamine deficiency in experimental animals requires at least 615 ppm of sulfur dioxide in the whole diet. Even if the sulfur dioxide content of dried fruits were 3,000 ppm the daily human diet would have to contain about 20% dried fruits to produce thiamine deficiency. Such a daily diet would not be feasible, because of undesirable laxative action.

BOOK REVIEWS

PROTEIN AND AMINO ACID REQUIREMENTS IN EARLY LIFE, L. Emmett Holt, Jr., Paul Györgyi, Edward L. Pratt, Selma E. Snyderman, and William M. Wallace, New York University Press, New York, 1960. VII, 63 pp. Price $1.00.

The material presented was largely assembled by the Committee on Nutrition of the American Pediatries Society. The authors are recognized for their research accomplishments in the field of protein nutrition, and for their efforts to apply the findings of modern nutrition research to food problems encountered by the pediatrician in his practice.

The objective of the book is implied in a quotation on a fly leaf: "There is probably no field of human thought in which sentiment and prejudice take the place of sound judgment and logical thinking so completely as dieteties." The authors seek to establish bases for nutritional practice in pediatries in terms of present-day concepts of nutrition and the research back of these concepts. Perhaps the best measure of their success is the impression left with the reader that dieteties has its roots deep in scientific principles. The physician will find answers to questions he may have in regard to the feeding of his patients, and persons outside the medical profession—researchers, teachers, dietitians, public-health workers, and food technologists—will also welcome the views of these authors on perplexing problems.

The various chapters deal with topics usually considered in evaluating protein needs of any age group.

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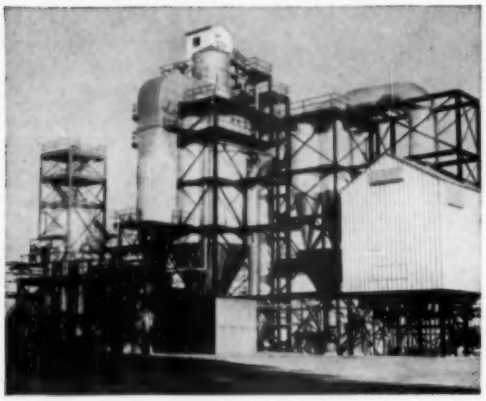

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It is an excellent summary of today's knowledge of protein nutrition and of the problems that baffle and intrigue the nutrition research worker. And the reader cannot but be aware of the many gaps in knowledge that exist today.

The importance the authors attach to understanding basic nutrition facts is found in their discussion of topics like the following: What is the nature of the growth phenomenon? What is the significance of nitrogen retention? What do we mean by protein reserves? Is the term "labile nitrogen" better? What is the picture of protein deficiency? What is the status of information about the protein requirement? What basic theoretical assumptions underlie evaluation of the protein requirement? How is the standard reference protein used in the study of protein requirements? What is meant by "factors of safety"? What are the problems in measuring protein quality? What is the influence of technological procedures used in the food industry on the quality of protein products? Why is the establishment of amino acid requirements of only limited practical significance? What is the meaning of amino acid imbalance? Of toxicity of specific amino acid excess? What are the long-term effects of high-protein diets? How does disease affect the protein metabolism?

In view of the findings of research in these various areas, the authors emphasize the need for more rather than less basic investigation of the characteristics of protein metabolism and of individual amino acids. But knowledge that will lead to the answer to practical questions must also be expanded. For instance, the gap in precise information on minimal requirements of proteins and amino acids up to adult life must be filled; these requirements must be studied in relation to protein-sparing components of the diet and to the amino acid patterns; methods for measuring protein adequacy must be refined; the optimum dietary amino acid pattern for health needs careful definition; the desirability of feeding protein above the minimal level needs careful study; and the relative importance of amino acid balance in the diets of people should be evaluated.

The authors show how nutrition research becomes entangled in problems of national and international importance. Is industry, for example, justified in unqualified recommendation of the use of high-protein diets? "Protein content and quality have become gimmicks in the food industry." To what extent is fortification of food with amino acids justified in underdeveloped countries? In well-developed countries? What is the responsibility of industry in the operation? Can large-scale low-cost production of the amino acids needed for use in an underdeveloped country be obtained only by exploitation of the idea in a well-developed country? As far as the United States is concerned, the authors agree with the stand taken by the Food and Nutrition Board that food additives are justified only when their incorporation into the food supply is likely to produce a public-health benefit. There is as yet "no evidence that specific amino acid deficiencies are a problem in the

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United States." Neither is there any "evidence of any general need for amino acid fortification."

The authors write further: "The possible danger of amino acid supplementation by the induction of an amino acid imbalance does not seem to be a serious one in a country like the United States with an abundant protein supply, for imbalances are readily produced only on low protein intakes. The objection to amino acid supplementation, apart from the fact that the need for it cannot be shown, lies in the fact that when promoted industrially it tends to foster food faddism and food nuerosis, a problem with which the pediatrician is only too familiar."

PEARL SWANSON

LIPIDE METABOLISM. By Konrad Bloch. John Wiley & Sons, Inc., New York. 1960, XIII, 411 pp. Price $10.50.

This book is a companion volume to Hanahan's *Lipide Chemistry*, which also appeared in 1960 (soon to be reviewed in FOOD TECHNOLOGY). The almost explosively rapid development of research knowledge in the field of lipide metabolism is reviewed and brought up to about the middle of 1959 by a group of authorities. The chapters and authors are: Enzymatic mechanisms of fatty acid oxidation and synthesis, Green and Wakil; Metabolism of the unsaturated fatty acids, Mead; Metabolism and function of phosphatides, Rossiter and Strickland; Metabolism of glycerides, Borgström; Lipolytic enzymes, Kates; Hormonal regulation of fatty acid metabolism, Langdon; Formation and metabolism of bile acids, Bergström, Danielsson, and Samuelsson; and Chemistry and metabolism of bacterial lipides, Asselineau and Lederer.

The approach is strictly biochemical, with some overtones of medicine in the chapter on hormones and fat metabolism. Biosynthetic and degradative pathways are spelled out in detail for fatty acids, glycerides, phospholipides, sterols, and bile acids. Nutritional aspects of lipide metabolism are not discussed. Thus, there is no reference to the vast literature that continues to accumulate on the meaningfulness or lack thereof of cholesterol or lipoprotein blood levels for heart disease.

Most food technologists will not be able to use this excellent compendium in their daily work. Nevertheless the future of food science compels a continuing awareness of the trends of biochemical research. This book makes available the information on lipide metabolism necessary for this purpose.

The book is pleasingly free of misprints and errors, well-printed, and extremely well documented, but the index is inadequate. The reader would do better to consult the longer and more complete table of contents in order to locate items of interest to him.

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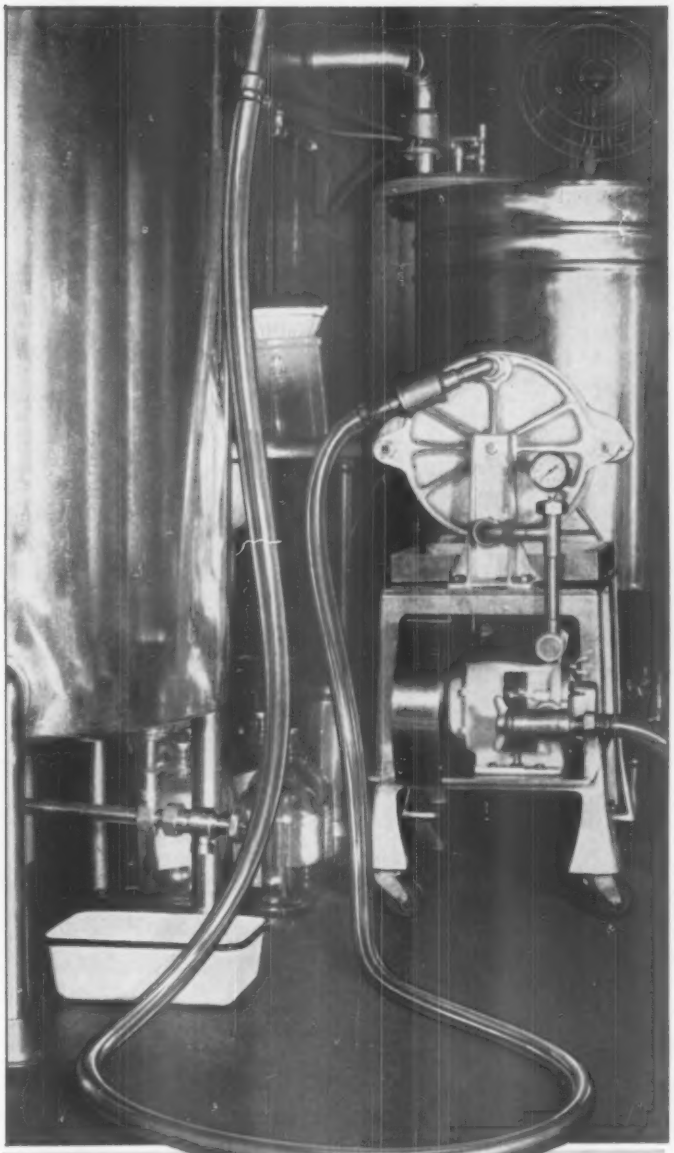

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PURE FOOD AND PURE FOOD LEGISLATION, Edited by A. J. Amos, B.Sc., Ph.D., F.R.I.C., London, Butterworths, 1960.

During the early 1800's F. C. Accum, a zealous young chemist, and later A. H. Hassall, an expert microscopist as well as a chemist, persistently applied their skills to exposing the scandalous extent to which foods were adulterated in the London of their day. Though they had no official capacity, they even revealed the names and addresses of the purveyors of these foods, and in this respect were more daring than some of the contemporary crusading scientists, who preferred anonymity. As a result of Hassall's disclosures, Wakeley, then editor of *The Lancet*, appointed him to head that medical journal's Analytical and Sanitary Commission, under whose auspices the investigations were extended. The interest of the British Parliament in the published findings led to enactment, in 1860, of the first comprehensive pure-food law in the English-speaking world, known as "An Act for Preventing the Adulteration of Articles of Food or Drink." To commemorate this event the Food Law Centenary was celebrated in London last September. In addition to food law-enforcement officials from various points of the world, it attracted many outstanding leaders in the areas of public health, nutrition, and food technology.

The present volume, ably edited by the chairman of the Organizing Committee, is composed of the eight papers and the discussions presented during the formal part of the proceedings. Dr. J. H. Hamence, then President Elect of the Association of Public Analysts, opened the conference with a historical account of the Act of 1860.

Speaking in behalf of the food industries, D. E. B. Hughes, of J. Lyons & Company, Ltd., describes the impact of this and subsequent legislation on present standards of quality control designed to ensure the cleanliness, wholesomeness, and safety of processed foods. Dr. Norman C. Wright, Deputy Director General of the Food Agricultural Organization, describes the international aspects of pure-food legislation not only in the United Nations agencies but also in various European and Latin-American organizations. He discusses the problem of establishing international agreement or at least some degree of uniformity in standards of identity of foods and in permissive lists of food additives.

Dr. C. A. Morrell, Director of Food and Drug Directorate for Canada, Dr. F. H. Reuter, Associate Professor of Food Technology of the University of New South Wales, and Dr. G. P. Larrick, U. S. Commissioner of Food and Drugs, report on various aspects of food law enforcement in their respective countries. Of especial interest to those concerned with federal vs. state controls is Professor Reuter's detailed account of the efforts to unify pure-food legislation between the States and the Commonwealth of Australia, which involved the active participation of the Council of Australian Food Technology Associations. Commissioner Larrick, like several other speakers, draws attention to some of the difficulties connected with enforcement of the new laws regulating pesticidal residues and food additives, particularly when suitable analytical methods are lacking.

The compilation of papers concludes with a plea by Professor Alastair C. Frazer, University of Birmingham, for better integration of food research having in mind the nutritional needs not only of populations subsisting on marginal or submarginal diets but those depending to a large extent on processed foods, citing flour as an example. Certainly borne out by recent experience in the U. S. is his observation that "If legislation runs ahead of available facts, difficulties are bound to arise."

The governmental and scientific bodies that sponsored the Food Law Centenary are to be commended for focusing attention on this historic landmark and for providing the opportunity for an international exchange of experience among those dedicated to protecting and improving man's food. It is gratifying that the addresses of the distinguished participants in the Conference have been recorded for the benefit of present and future food scientists, educators, and administrators.

BERNARD L. OSER

FOODS WITHOUT FADS. E. W. McHenry, J. B. Lippincott Company, East Washington Square, Philadelphia, Pa., $3.50.

The book, designed to bring food facts to the public, is simply written for the layman. It deals with the basic foods and their contributions to a balanced diet. Dr. McHenry has discussed good nutrition and the selection of foods taking into consideration factors such as economic, customs, and religious precepts in the foundation of balanced diets. Special food problems encountered in the obese, the young, the aged, and in disease are considered. The pros and cons of vitamin supplementation are discussed in the chapter "Food or Pills." A warning is given on the danger of large excesses of vitamins A and D. Dr. McHenry feels that, in general, "It pays to eat wisely, and not spend money on pills." A balanced diet contains all of the essential nutrients except vitamin D, which is needed by babies, children, adolescents, and expectant mothers during the second half of pregnancy.

The chapter "Food Nonsense is a Nuisance" deals with fad diets compounded by "alleged" experts. Many of these experts have had little or no training in nutrition at the university level.

Dr. McHenry concludes that "Helpful meals help to attain and maintain health at any age. Food intake is not the only factor involved in the maintenance of health: it is one factor which we don't need to neglect. Good food selection means good meals. Good meals are worth eating." We agree.

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This should eliminate several, and when your decision is final, submit your Nomination in letter form including the following:

A. Name of Company or Institution
B. Name of Product and/or Process
C. Description of Product or Process
D. Statement or reason for considering this a meritorious achievement.
E. Statement listing individuals chiefly responsible for the achievement and their individual contributions
F. Statement indicating the time and extent of commercial utilization

Nominations must be limited to an absolute maximum of three single-spaced typewritten pages.

The Company or Institution winning the Food Technology Industrial Achievement Award will be presented a bronze Plaque plus, insofar as possible, public recognition at the time of presentation during the 22nd IFT Annual Meeting at Miami Beach in 1962. Furthermore, the individual(s) chiefly responsible for the product or process, either through basic research or development, will each be presented with an engrossed plaque and be given public recognition for his achievements.

While the Award is available each year, such high standards of merit are expected that the following is included in the rules governing it: "It is, therefore, not anticipated that a suitable meritorious subject will be found every year. It is accordingly the prerogative of the Awards Jury to pass or postpone the Award whenever in the judgment of the Jury none of the nominations fully meet the high standards of achievement for which this Award is established."

Upon completion of your Nomination, submit it to:

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DEADLINE FOR RECEIPT—December 1, 1961

Coming Events

1961

Sept. 19–22	Food Science Conference, C.S.I.R.O., North Ryde, N.S.W., Australia
Sept. 19–27	International Conference on Fish in Nutrition, Washington, D. C.
Sept. 28–30	American Hotel Association, Washington, D. C.
Oct. 7–12	Baking Industry Exposition, Convention Hall, Atlantic City
Oct. 15–18	National Association of Food Chains, Palmer House, Chicago, Illinois
Oct. 18–20	National Packaging Forum of Packaging Institute, Biltmore Hotel, New York City
Oct. 16–19	Second International Congress of the International Organization for Vacuum Science and Technology, Washington, D. C.
Oct. 19-20	European Federation of Chemical Engineering Symposium on Food Technology, Frankfurt/Main, Germany
Oct. 19–20	Symposium on Freeze-drying, London
Oct. 23–28	First Inter-American Congress of Chemical Engineering, San Juan, Puerto Rico (write Institute of Chemical Engineers of Puerto Rico, P. O. Box 47, Rio Piedras, Puerto Rico)
Oct. 24–27	American Dietetic Association, Jefferson Hotel, St. Louis
Oct. 28–31	National Automatic Merchandising Assn. Convention and Exhibit, Chicago, Illinois
Nov. 5–8	National Frozen Food Association, Bal Harbour and Americana Hotels, Miami Beach
Nov. 6–10	National Hotel Exposition, Coliseum, New York City
Nov. 12–15	Air Conditioning and Refrigeration Institute, Homestead Hotel, Hot Springs
Nov. 15	British National Association of Wholesale Distributors of Frozen Foods—Annual Lunch, Trocadero, London
Nov. 27–Dec. 1	28th Exposition of Chemical Industries, New York Coliseum
Dec. 1-3	National Automatic Merchandising Assn. Western Conference and Exhibit, Los Angeles, California

1962

Feb. 9–11	Institute of American Poultry Industries' 33rd Annual Fact Finding Conference; Municipal Auditorium, Kansas City, Missouri
May 10–12	International Symposium on Food Protection, Department of Dairy and Food Industry, Iowa State University, Ames, Iowa
June 10–14	**Twenty-second Annual Meeting of the Institute of Food Technologists, Fontainebleau Hotel, Eden Roc Hotel, Miami Beach, Florida**

An open invitation is extended to readers of FOOD TECHNOLOGY to send in to the Editorial Office, P. O. Box 164, Davis, California, notices of annual or national meetings of interest to food technologists.

People

United Fruit Company has named **Howard L. Stier** to the newly created position of Director of Quality Control, with headquarters at the Company's General Office, in Boston. A native of Delmar, Delaware, with degrees in Horticulture and Plant Physiology from the University of Maryland, Dr. Stier has followed a career as a research administrator, statistician, and educator. He comes to this new assignment from the National Canners Association, where, for the past ten years, he was Director of the Division of Statistics, which involved economic and marketing studies, quality control, and the analysis of experimental data. While at NCA he also served as a professorial lecturer in managerial statistics and quality control at George Washington University. Prior to his association with National Canners, Dr. Stier was Professor of Marketing and head of the department at the University of Maryland, College of Agriculture.

H. L. Stier

D. M. Doty

C. F Niven, Jr.

Promotion of two long-time members of the staff to top positions at the American Meat Institute Foundation has been announced recently by **Homer R. Davison,** President of the Foundation, and Chancellor **George W. Beadle,** University of Chicago.

Dr. Delbert M. Doty, a member of the AMIF staff since 1948 and an associate director of research and education there since 1956, has been named AMIF Director of Research and Education. In his new assignment, Dr. Doty assumes administrative direction of the AMIF. Dr. Doty had all his academic training at Purdue University, from which he received his Ph.D. degree in agricultural chemistry. Before joining the Foundation staff in 1948 as chief of the division of analytical and physical chemistry, he served in various positions on the staff of the Purdue Department of Agricultural Chemistry, including duties as acting head as well as associate professor and assistant chief of the department.

Dr. Charles F. Niven, Jr., is the new Scientific Director. Dr. Niven became chief of the AMIF division of bacteriology when research operations began at the Foundation

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in 1947, and was appointed an associate director in 1956. He will supervise the activities of a staff of 40 scientists. Dr. Niven did his undergraduate work at the University of Arkansas and received his Ph.D. degree in bacteriology from Cornell University. After several years on the faculty at Cornell he was bacteriologist for Hiram Walker & Sons, and joined the research staff of the American Meat Institute in 1946. Since 1953 Dr. Niven has served successively as assistant and then associate director of research.

Two scientists have been added to the Lipton Research Organization, according to **Dr. Roy E. Morse,** Director of Technical Research for Thomas J. Lipton, Inc. **Francis Zientara,** at present working for his master's degree at Brooklyn Polytechnic Institute, comes to Lipton from Schwartz Laboratories' brewing technology section. He is a graduate of Fordham University with a B.S. degree in chemistry and has taken additional courses at Columbia University. **Warren Wong,** formerly in the biochemistry and flavor section of General Foods research department, has been assigned to carbohydrate studies. Mr. Wong holds a B.A. degree in chemistry from Rutgers University.

Valentine H. Fischer, vice-president of Dodge & Olcott, Inc., announces his retirement after 44 years with the firm. Mr. Fischer is an authority and prominent world trader in the essential oils and spice markets. He is former president of the E.O.A. and is an active member of the IFT and other allied associations.

V. H. Fischer

Ernest Wolf has been appointed Development Manager of Charles H. Kline & Company, Inc., Pompton Lakes, New Jersey. He was formerly manager of chemical sales for the International Division of Baxter Laboratories, Inc., and coordinator of commercial development for Baxter's Wallerstein Company division. Before coming to Baxter in 1959, he was with the Olin Mathieson Chemical Corporation, working in areas of chemical research and commercial development. Mr. Wolf received a B.S. degree from Queens College, Flushing, New York, an M.S. degree in organic chemistry at Purdue University, and an M.B.A. degree from the University of Buffalo.

Ernest Wolf

H. R. Goff, President of James Dole Engineering Company, recently announced the election of **Dr. Roger Lueck** to the Board of Directors. Dr. Lueck, a Charter Professional Member of the IFT, is Vice-President of American Can Company in charge of research, machinery development and new products, and has his office in New York City.

Industry News

BRITISH KIPPERS TO MOSCOW

A refrigerated truck containing 2,000 pairs of kippers drove the 2,000 miles from London to Moscow to provide the recent British Trade Fair in Moscow with this British specialty. This delivery was part of a promotion to open up the Soviet food market for this particular item. It was sampled by officials of the Soviet Food Ministry.

LEAF PROTEIN MILK PLANT IN JAMAICA

A machine for extraction of protein from the leaves of tropical plants is being constructed in Jamaica, West Indies. It is the result of more than 20 years of research on leaf protein extraction, primarily by Dr. A. J. Pirie, Rothumsted Research Institute, U. K.

The extracted protein is to be used as an additive in both human food and animal feeds. The plant is being erected by the University College of the West Indies under supervision of the Scientific Research Council of Jamaica.

The unit is designed to be mobile so as to take maximum advantage of the varying tropical vegetation of the 4,400-square-mile island. It will produce 20–80 lb of protein from one ton of leaves or grass in an hour.

STREAMLINED JAM PROCESSING IN U.K.

A number of operations have been combined by the British machine industry of Robert Kellie and Son, Ltd., in order to facilitate automation in jam production. The key machine in the finishing line is the Kellie **Hydroseal,** a sterilizer and cooler with an intake of 120 jars per minute and a holding capacity of 4,350 jars. The combined sterilization, formation of vacuum, and cooling takes 33 minutes. The Hydroseal is lined up with filling and capping machines at the finishing end with a jar-drying conveyor and a labeling machine. Such a processing line allows a factory to have cartons of jam ready for dispatch 55 minutes after the fresh fruit is received. (Food Manufacture **36** (5), 197–198.)

GAS STORAGE FOR BLACK CURRANTS

Black currant juice has become, next to apple juice, the dominant fruit juice of Europe. This ex-

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CORN SYRUP SOLIDS are widely used in ice cream, baked goods, frozen fruits, dry food mixes, jams, jellies, table ready and canned meats, to mention just a few.

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plains extensive studies made on the handling of this particular berry. Storage tests under varying conditions have recently been reported on by the Ditton Laboratory, England. The degree of mold contamination was assessed. Results indicated that a high concentration of carbon dioxide in the storage atmosphere greatly retarded the development of fungal organisms. Black currant could be stored satisfactorily for at least 8 weeks under such conditions. By such means, it is therefore possible to lengthen the period during which fresh black currants are available to jam and juice manufacturers.

A DANISH PECTIN FACTORY—WORLD'S LARGEST EXPORTER

A/S Københavns Pektinfabrik started in 1934 as a fruit preserves factory in Copenhagen, pectin for manufacture being imported from the U.S.A. and Germany. Two years later, however, the company decided to produce its own pectin, and other plants soon became buyers. In the post-war period a substantial market was created abroad for this Danish pectin. The factory moved to Lille Skensved, near Køge, and today the company is the largest pectin exporter in the world, 95% of the production being exported. The U.S.A., although accounting for about half of the world's large pectin factories, is nevertheless the greatest buyer of Danish pectin.

Jam and confectionery factories take about 90% of the world production of pectin. It is also used, among other things, as a stabilizer in ice cream.

The Danish pectin plant is constantly carrying on research to improve production methods. A complete pilot plant has been established, permitting the testing of all phases of the manufacturing processes. A series of experiments with a new centrifuging and filtering technique based on a fully automatic process is just reported as concluded. A new steam plant, fractionating column, and vacuum evaporation system are being installed in the plant after careful pretesting.

New laboratories are now being built, and the research teams expanded to develop new products and production methods as well as carrying out basic research. (Food Manufacture **36** (6), 250–256.)

BRITISH BAKING RESEARCH EXPANDED

The British Baking Industry inaugurated an added wing to its research laboratories at Chorleywood in May of this year. The new building consists of a two-story building. Its main features are a pilot-scale biscuit bakery and several new devices for applied research. Among these are a forced convection traveling oven and a rotary biscuit cutter. The biscuit section is thus equipped to study most phases of the manufacture of both hard and soft biscuits.

A Buhler experimental mill enables the institute to mill its own samples of flour from selected wheats and thereby tackle problems concerned with the composition of the lipid and protein fraction of flour. A new microbiology laboratory in this wing is provided with separate rooms for sterilization. This section is provided with dust-free atmosphere, through special filtering equipment. The old part of the building houses the sections for organic and physical chemistry. The new wing also accommodates special offices devoted to the application of scientific and technical findings in the baking industry. The task is also to spot and assess problems of the industry that require research attention.

REFRIGERATION PROGRAM FOR YUGOSLAVIA

A refrigeration program, together with details of required equipment, has been announced in Yugoslavia. The present capacity for refrigerated food storage, in 1,000-ton units, is for Serbia 24.4, Croatia 9.1, Slovenia 5.0, Bosnia and Herzogovina 4.76, Macedonia 7.5, Montenegro 0.7; a total of 51,540 tons or 209,000 m.^{3}, plus a further 71,000 m.^{3} for the beer and wine industries. Of the total amount of foodstuffs produced in 1965, about one fourth (2,230,000 tons) will need refrigeration, i.e., a cold-storage capacity of 820,000 m.^{3} is needed. This means a considerable expansion in the presently available refrigerated storage space, which does not meet the needs. (Simončič, J., 1960, Refrigeration in Yugoslavia (In German) *Kältetechnik* No. 11, 339–340.)

The development of a new industrial **rust and scale remover** and stainless-steel cleaner is announced by **Berman Chemical Company.** Known as "Bull Frog" Scale Off No. 104, the product comes in dry powder form and is manufactured for the cleaning of kettles, cookers, evaporators, hoppers, and food pipelines. Literature may be obtained from Berman Chemical Company, 1795 No. 12th Street, Toledo, Ohio.

Effective at the close of business July 12, 1961, the **C. H. Musselman Company** merged into the **Pet Milk Company.** With the addition of Musselman, Pet Milk Company announces that it will operate more than 75 plants throughout the nation and in Canada, and have an annual sales volume in excess of $220,000,000. Pet now has four major operating divisions: The C. H. Musselman Company Division, Food Products Division, Pet Dairy Products Company Division, and Pet Milk Company (Canada), Ltd.

Durkee Famous Foods announces that an agreement for development

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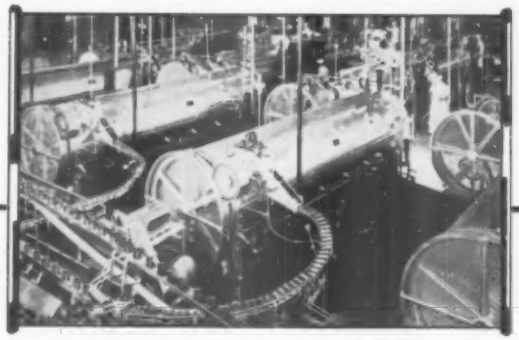

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and marketing of a **new safflower margarine oil** has been entered into by **The Glidden Company** and the **Pacific Vegetable Oil Corporation,** San Francisco. Pacific Vegetable Oil will provide an assured source of safflower oil, and Durkee will produce and market the margarine oil, the announcement said.

National Honey Week will be observed throughout the United States and Canada from October 23 to 29. Stores will be stocked with comb honey, liquid or extracted honey in various sizes of containers, finely crystallized honey of creamy consisteney, cut comb and chunk honey. Theme for the week is "Enjoy Honey Every Day."

A new 48-page booklet, describing the **properties and uses of organic acids and anhydrides,** has been published by **Union Carbide Chemicals Company**. It contains data on acetic, propionic, butyric, 2-ethylbutyric, 2-ethylhexoic, acrylic, sorbic, valeric, and isodecanoic acids; acetic, propionic, and butyric anhydrides. Included is information on physical properties; constant-boiling mixtures; specification limits, test methods; storage, handling, and shipping; toxicological properties; and selected literature references. For further information, write Union Carbide Chemicals Company, 270 Park Avenue, New York 17, New York.

Archibald & Kendall Inc. announces the merger of **Kearns & Smith Spice Company**, Chicago, with Archibald & Kendall Inc., New York. No changes in the management and policies of the two merged companies are contemplated, and they will continue distribution of their products under their brand names.

Dimensional stability, reduced cost, and lower density are the features of a **new urethane insulating foam** described in a bulletin published by **Nopco Chemical Company.** The bulletin describes the material, provides information on advantages, mixing, and K-factors, and illustrates results of low-temperature tests on conventional foam and the new formulation. It also furnishes a listing of physical properties including densities, strengths, thermal conductivity, and dimensional stability figures. Copies may be obtained by writing Plastics Division, Nopco Chemical Company, 175 Schuyler Avenue, North Arlington, New Jersey.

A new 4-page folder covering the complete line of **Blower Units** manufactured by **Systems Engineering and Manufacturing Company** has just been released. The literature covers both low- and high-pressure models, including schematic drawings, specifications, and standard model designations, together with operating and maintenance instructions. Write Systems Engineering and Manufacturing Company, Inc., P. O. Box 7634, Houston 7, Texas.

Certified Midnite Black Shade, the first true black color with U. S. Food & Drug Administration certification, has been developed by the **Bates Chemical Company** for candy manufacturers and other food-color users, Guy C. Bates, the firm's president, announced recently. The new Bates mixture, composed of FD&C primary colors, is water-soluble. For samples and additional information, write Bates Chemical Company, Lansdowne, Pennsylvania.

Elion Instruments, Inc., has announced the completion of the **DEM No. 301 Electron Microanalyzer.** The unit was developed to examine and identify submicro-

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scopic particles as small as one-tenth of a micron in diameter. The new microanalyzer is said to be the largest and most important product yet developed by the firm. It is currently priced at $68,000. For additional information, write Elion Instruments, Inc., 430 Buckley Street, Bristol, Pennsylvania.

Niagara Blower Company announces the development of **"No-Frost" Refrigerating Units,** designed for use in automatic continuous-production chilled-air quick-freezing of food products and prepared food specialties. Refrigerant temperatures may be minus 35°F or lower, and multiple-unit installations are sized to fit any freezing operation, according to the announcement. For further information, write Niagara Blower Company, 405 Lexington Avenue, New York.

Armour & Co. recently announced plans to build a food research laboratory in Oak Brook, Illinois, a Chicago suburb. The facility will include an analytical laboratory, taste testing laboratory, packaging materials section, technical library, three pilot plants and research bakery. It is anticipated that one hundred scientists and technicians will work toward developing new and improved meat, dairy, poultry, and shortening products. Research will range from basic investigation of the chemistry of proteins and fats to pilot plant studies of food manufacturing processes. Occupancy is scheduled in about a year.

Dr. Charles Edward North, bacteriologist and dairy industry sanitation pioneer, died in Montelair, N. J. July 27th. He would have been 92 years old the following day. Dr. North's early work in the dairy industry aided in establishing sanitation standards credited with virtually eliminating infant mortality due to raw milk. His work was said to have revolutionized the standards of sanitation in that field and to have contributed to modern milk supply purification techniques. Survivors include his widow, Amelia; a son, Charles, and four daughters.

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Reply directly to ad BY BOX NUMBER (where indicated) via: Executive Secretary, Institute of Food Technologists, 176 West Adams Street, Chicago 3, Illinois.

MARKETING MAN—15 years in Market Development, Market Research, Promotion and Sale of food ingredients and related materials. Chemical degree and administrative experience. Now in organic chemicals, want out. Please write BOX 892.

AVAILABLE: Food Technologist, B.S. Purdue 1948. Some graduate work. Experience — canning, carbonated beverages and dairy products. Desire Chicago location. REPLY BOX 901.

Mech. Engineer wants position preferably at management level. 14 yrs. in the milk and food business, with extensive knowledge on dehydration, complete plant layouts, production as well as auxiliary equipment such as refrigeration boilers, turbines and diesel motors. REPLY BOX 900.

B.S. degree in Chemistry and Mathematics. 8 yrs. exp. in Statistical Quality Control and as Governmental Food Control Chemist. Some experience with meat. Able to handle correspondence and personnel. Desire position in production, technical service or quality control in South or Southwest. Salary $7,000. REPLY BOX 857.

Responsible position desired in areas of technical sales, sales development, sales service, or product development to food processors. Excellent record covering fifteen years experience in above fields. Major experience in frozen desserts, flavors, and syrups. REPLY BOX 899.

AVAILABLE: Operations Manager, Food Technologist, 40, B.S., MBA. 13 yrs. proven exp. in line management, creative product development, Research and Quality Control. Ability to run an entire food processing operation, direct new product development, institute cost reduction studies and get results, and coordinate consultative services for industrial, merchandising, and procurement staffs. Presently employed as Operations Manager for plant of 50 people. Excellent references. Salary $14,500. REPLY BOX 894.

AVAILABLE: Food Technologist, Executive, Versatile, M.S. Experience with Frozen Prepared Foods, Dairy Products, Pressurized Foods, Commissary. Age 30. Married. REPLY BOX 895.

WANTED: College graduate in bacteriology or food technologist with bacteriological interest for research position in canning industry. Location: Washington, D. C. Travel involved infrequently. Experience with food not necessarily essential for applicant. REPLY BOX 896.

POSITIONS AVAILABLE

Reply directly to ads BY BOX NUMBER (where indicated), including the company name of your current employer, via: Executive Secretary, Institute of Food Technologists, 176 West Adams Street, Chicago 3, Illinois.

ASSISTANT
PRODUCTION SUPERINTENDENT

Large food company located in Central America has opening for Assistant Production Superintendent of canning operation. Requires degree in Food Technology or Engineering and production experience with heat processed foods. Good knowledge of Spanish essential. REPLY BOX 897.

RESEARCH CHEMISTS MILLING INDUSTRY

Organic chemist; or biochemist; Ph.D. degree, recent grad. for basic work on the fractionation and identification of flour constituents.

Food Technologist; B.S. degree, recent graduate or up to 3 years experience for development work on dessert products.

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TECHNICAL SALES REPRESENTATIVE—Position available on East Coast for a Technical Sales Representative to contact ingredient users of manufactured dairy products. We would like a man with some previous experience and a B.S. in Food Technology or Dairy Industry. Salary $6,000 to $7,500. Send résumé. REPLY BOX 893.

DIRECTOR RESEARCH AND DEVELOPMENT (FOODS)

Sizable Midwest food manufacturer and distributor needs a strong technical executive to coordinate and administer research, development, and quality control for the corporation. Covers new product development, testing, and formula modifications for existing food products. We require experience directing product development for a good company of medium size, or assistant director in a large company. Must be able to work effectively with both merchandise managers and operating personnel, and to realistically appraise products and quality levels desired by customers. Advanced degree in Food Technology is a must. Salary and bonus can be made attractive to you. Please give full information about your background, in confidence. REPLY BOX 903.

WANTED
Potential Sales Manager

Leading Midwestern corn mill seeks outstanding young salesman with management potential. Successful applicant will start in the field, take over full responsibility for sales of company products to packaged food industry as soon as he has demonstrated necessary qualifications. College degree, knowledge of food industry and fine previous sales record are essential. Submit complete résumé. REPLY BOX 902.

FOOD TECHNOLOGIST

Unusual opportunity for responsible positions in food process research and equipment development with established international manufacturer of food processing and packaging machinery in Northern California.

Prefer man having Ph.D. or M.S. with 5–8 years of applicable experience including some supervisory responsibilities. Knowledge of conventional and new food preservation techniques and space feeding problems is desirable. Work will include chemical, physical, bacteriological and biological problems of taste, appearance, spoilage, etc.

Reply should specify education, experience, responsibilities and salary history. REPLY BOX 898.

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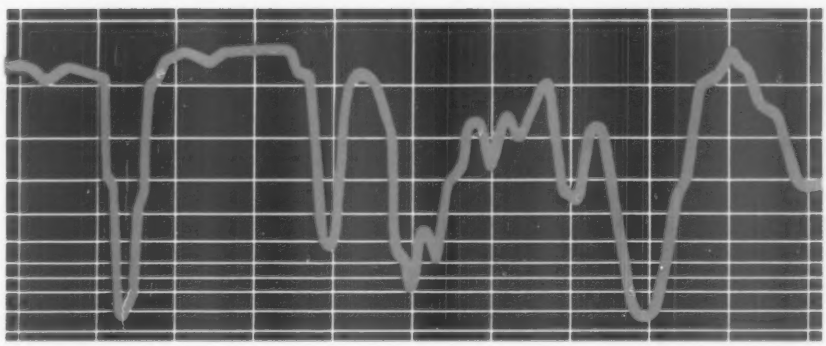

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